Supporting Information

Cobalt(II)-Catalyzed *peri*-C(sp^2)–H Selective Hydroxylation of Naphthalene Monoimides

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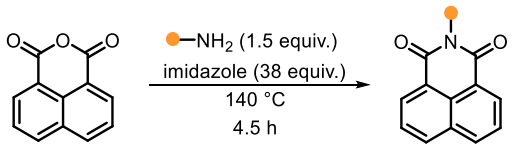
1. Materials and Methods

General Methods: All commercially available compounds were used without purification. Unless otherwise noted, all reactions were performed in oven-dried glassware. All solvents used in the reactions were purified before use. Tetrahydrofuran and toluene were distilled from sodium and benzophenone, whereas dry dichloromethane and dichloroethane were distilled from CaH_2.[1] Petroleum ether with a boiling range of 40–60 °C was used. ^{1}H, ^{13}C and ^{19}F NMR: Recorded on Bruker Avance III 400 MHz NMR Spectrometer, Bruker Avance III 500 MHz NMR Spectrometer and Bruker Avance III 700 MHz NMR Spectrometer; spectra were recorded at 295 K in DMSO-*d*$_6$ and $CDCl_3$; chemical shifts were calibrated to the residual proton and carbon resonance of the solvent: DMSO-*d*$_6$ (^{1}H δ 2.50; ^{13}C δ 39.52) and $CDCl_3$ (^{1}H δ 7.25; ^{13}C δ 77.0). HRMS: Bruker Daltonics MicroTOF Q-II with electron spray ionization (ESI) and Atmospheric Pressure Chemical Ionization (APCI). IR spectra were recorded on a FT-IR Spectrometer System (PerkinElmer Spectrum Two) and are reported in the frequency of absorption (cm^{-1}). Single Crystal X-ray Diffraction data were collected on a Bruker D8 Venture diffractometer equipped with Photon-III detector using monochromated Mo Kα radiation (λ = 0.71073 Å) at 140 K using an Oxford cryostream low-temperature device. Optical rotations $[\alpha]_D$ were measured at the sodium D line on a Rudolph Research Analytical Autopol II Automatic Polarimeter and the concentrations *c* are given in g/100 mL.

Steady-State Absorption and Fluorescence Measurements: A Cary 100 UV-vis spectrophotometer from Agilent Technologies was used for the measurements of steady-state absorption spectra for the compounds **9t** and **11** in all the solvents (DCM, *n*-hexane, Ethyl acetate, acetonitrile, methanol and toluene) with the proper baseline correction. For the emission spectra measurements, a Fluorolog 3-111 spectrophotometer was used in all sample measurements. We used a 1 cm path length standard quartz cuvette for the measurements of steady-state absorption and emission spectra in all the cases. Both the excitation and emission slits for all the samples (**3t** and **11**) in all the solvents were kept at 2 nm each.

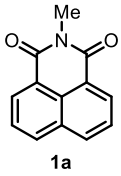
2. General Procedures and Analytical Data

Scheme S1: Synthesis of naphthalene monoimides (1a-1d, 1h & 1j).[2]

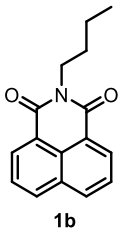


General Procedure: Naphthalene monoanhydride (NMA) (182 mg, 1 equiv., 0.92 mmol), amine (1.5 equiv., 1.38 mmol) and imidazole (2.38g, 38 equiv., 34.96 mmol) were taken in a pressure tube equipped with a stir bar. The tube was fitted with a Teflon screw cap under an argon flow. The reaction mixture was heated to 140 °C in silicone oil bath and allowed to stir for 4.5 h. Upon completion of the reaction, the reaction mixture was cooled to room temperature. The reaction mixture was diluted with DCM (75 mL) and this solution was washed with 2 M HCl (50 mL). The organic extract was dried with anhyd. Na_2SO_4, filtered and concentrated under reduced pressure. The resulting residue was purified by silica gel flash column chromatography eluting with EA:Hexane (2:98) to yield the desired product.

Analytical Data:

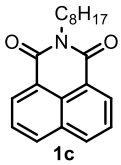


2-Methyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione:[2] Yield: 79% (153 mg); Physical appearance: White solid; TLC R_f 0.20 (Petroleum ether:Ethyl acetate, 1:1); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 8.50 (d, J = 7.2 Hz, 2H), 8.46 (d, J = 8.2 Hz, 2H), 7.88 (t, J = 7.7 Hz, 2H), 3.41 (s, 3H).



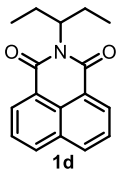
2-Butyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione:[2b] Yield: 90% (209 mg); Physical appearance: yellow solid; TLC R_f 0.5 (Petroleum ether:Ethyl acetate, 9:1); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.64 (d, J = 7.3 Hz, 2H), 8.24 (d, J = 8.2 Hz, 2H), 7.78 (t, J = 7.7 Hz, 2H), 4.22 (t, J = 7.5 Hz, 2H), 1.82 – 1.69 (m, 2H), 1.53 – 1.43 (m, 2H), 1.01 (t, J = 7.4 Hz, 3H).

2-Octyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione:[2] Yield: 80% (227 mg); Physical appearance: Bright yellow solid; TLC R_f 0.20 (Petroleum ether:Ethyl acetate, 9:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.60 (d, *J* = 7.3 Hz, 2H), 8.21 (d, *J* = 8.3 Hz, 2H), 7.75 (t, *J* = 7.7 Hz, 2H), 4.19 (t, *J* = 7.8 Hz, 2H), 1.79 – 1.69 (m, 2H), 1.49 – 1.27 (m, 10H), 0.92 – 0.85 (m, 3H).

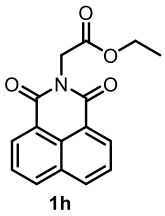


1c

2-(Pentan-3-yl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione:[2] Yield: 86% (211 mg); Physical appearance: White solid; TLC R_f 0.20 (Petroleum ether:Ethyl acetate, 19:1); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.60 (d, *J* = 7.3 Hz, 2H), 8.22 (dd, *J* = 8.3, 0.92, 2H), 7.78 (t, *J* = 7.7 Hz, 2H), 5.12 – 5.04 (m, 1H), 2.34 – 2.21 (m, 2H), 2.00 – 1.87 (m, 2H), 0.92 (t, *J* = 7.5 Hz, 6H).

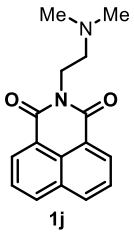


1d



1h

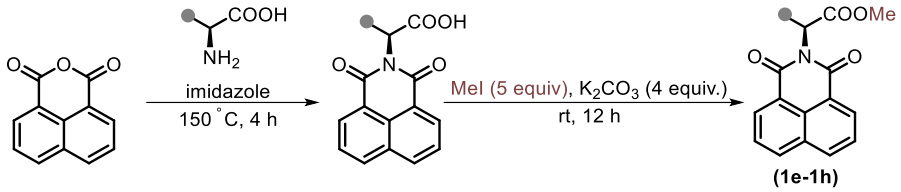
Ethyl 2-(1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)acetate: Yield: 56% (146 mg); Physical appearance: White solid; R_f 0.35 (EtOAc:Hex, 1:9); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.66 (d, *J* = 7.2 Hz, 2H), 8.28 (d, *J* = 8.2 Hz, 2H), 7.80 (t, *J* = 7.8 Hz, 2H), 4.98 (s, 2H), 4.28 (q, *J* = 7.2 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H).



1j

2-(2-(Dimethylamino)ethyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione: Yield: 43% (106 mg); Physical appearance: White solid; R_f 0.25 (100% EtOAc); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.63 (d, *J* = 7.3 Hz, 2H), 8.25 (d, *J* = 8.2 Hz, 2H), 7.78 (t, *J* = 7.8 Hz, 2H), 4.47 (t, *J* = 6.9 Hz, 2H), 2.96 (t, *J* = 6.9 Hz, 2H), 2.59 (s, 6H).

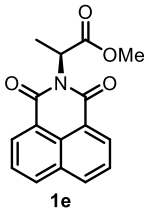
Scheme S2: Protection of the C-center of the coupled amino acids using methyl iodide (1e-1g).[3]



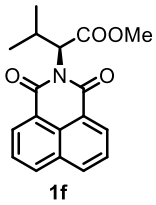
General Procedure: The crude product (1.1 mmol, 1 equiv.) formed in the previous step was dissolved in acetone (4 mL) in a 25 mL round-bottom flask equipped with a magnetic stir bar along with K_2CO_3 (608 g, 4.4 mmol, 4 equiv.). Methyl iodide (0.4 mL, 5.5 mmol, 5 equiv.) was added *via* a syringe. The reaction mixture was allowed to stir at room temperature for 12 h. The reaction mixture was then concentrated under reduced pressure. The resulting residue was purified using silica gel flash column chromatography (2:8, Ethyl Acetate: Hexane).

Analytical Data:

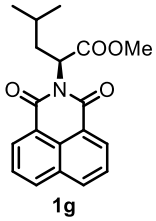
Methyl-*(S)*-2-(1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)propanoate:[2b] Yield: 72% (187 mg); Physical appearance: white solid; TLC R_f 0.45 (Petroleum ether:Ethyl acetate, 9:1); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.64 (d, *J* = 7.2 Hz, 2H), 8.27 (d, *J* = 8.1 Hz, 2H), 7.80 (t, *J* = 7.6 Hz, 2H), 5.80 (q, *J* = 6.7 Hz, 1H), 3.76 (s, 3H), 1.72 (d, *J* = 6.8 Hz, 3H); **$[\alpha]_D^{29.2}$** = –1.36 (*c* 0.33, $CHCl_3$).


1e

Methyl-*(S)*-2-(1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)-3-methylbutanoate:[2b] Yield: 40% (114 mg); Physical appearance: white solid; R_f 0.50 (1:9 EtOAc:Hex); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.65 (d, *J* = 7.3 Hz, 2H), 8.28 (d, *J* = 8.3 Hz, 2H), 7.81 (t, *J* = 7.8 Hz, 2H), 5.40 (d, *J* = 9.2 Hz, 1H), 3.71 (s, 3H), 2.94 – 2.85 (m, 1H), 1.34 (d, *J* = 6.5 Hz, 3H), 0.84 (d, *J* = 6.9 Hz, 3H); **$[\alpha]_D^{29.3}$** = –1.88 (*c* 0.33, $CHCl_3$).

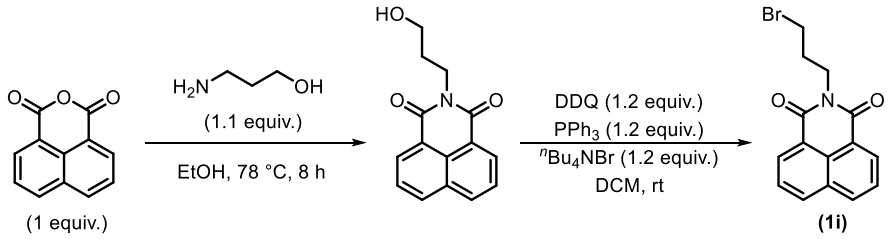

1f

Methyl-*(S)*-2-(1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)-4-methylpentanoate:[2b] Yield: 50% (149 mg); Physical appearance: white solid; R_f 0.55 (EtOAc:Hex, 1:9**); ^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.64 (d, *J* = 7.3 Hz, 2H), 8.27 (d, *J* = 8.2 Hz, 2H), 7.80 (t, *J* = 7.8 Hz, 2H), 5.82 (dd, *J* = 9.3, 4.9 Hz, 1H), 3.74 (s, 3H), 2.30 – 2.22 (m, 1H),


1g

2.18 - 2.10 (m, 1H), 1.65 – 1.59 (m, 1H), 1.04 (d, *J* = 6.5 Hz, 3H), 0.95 (d, *J* = 6.7 Hz, 3H); **$[\alpha]_D^{29.1}$** = –30.91 (*c* 0.33, $CHCl_3$).

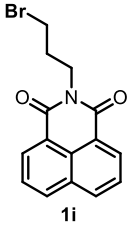
Scheme S3: Synthesis of naphthalene monoimides (1i).[3,4]



General Procedure: To a stirred solution of naphthalene monoanhydride (NMA) (1 g, 1 equiv., 5.07 mmol) in EtOH (15 mL) was added the corresponding primary amine (419 mg, 1.1 equiv., 5.60 mmol). The resulting mixture was heated at reflux temperature for 8 h and the progress was monitored by TLC. Upon completion, the reaction mixture was cooled to room temperature and concentrated under reduced pressure to give a solid. The crude product was washed with water and recrystallized from EtOH to give *N*-(hydroxyl)alkyl NMI in almost quantitative yield.

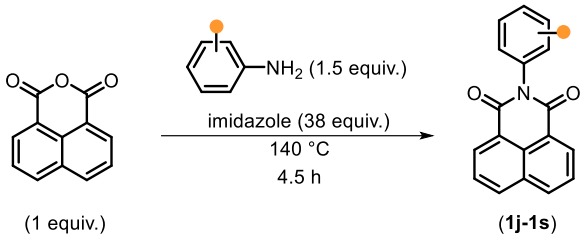
To a stirred solution of DDQ (227 mg, 1.2 equiv., 1.41 mmol) and PPh_3 (262.3 mg, 1.2 equiv., 1.41 mmol) in dry DCM (4 mL) was added tetrabutyl ammonium bromide (322.4 mg, 1.2 equiv., 1.41 mmol) at room temperature. The starting material *N*-(hydroxyl)alkyl NMI (300 mg, 1 equiv., 1.17 mmol) was the added to this mixture, which immediately turned the yellow colour of the reaction mixture to brown. The mixture poured onto a silica pad and eluted to yield the corresponding *N*-(bromo)alkyl NMI.

2-(3-Bromopropyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione:[3,4] Yield: 46% (171 mg);



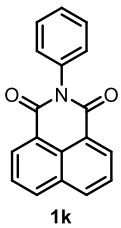
Physical appearance: White solid; TLC R_f 0.40 (Petroleum ether:Ethyl acetate, 9:1); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.65 (d, *J* = 7.2 Hz, 2H), 8.26 (d, *J* = 8.3 Hz, 2H), 7.80 (t, *J* = 7.8 Hz, 2H), 4.37 (t, *J* = 7.0 Hz, 2H), 3.53 (t, *J* = 6.9 Hz, 2H), 2.42 – 2.32 (m, 2H).

Scheme S4: Synthesis of naphthalene monoimides (1k-1t).[2]

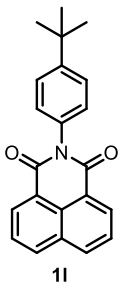


General Procedure: Naphthalene monoanhydride (NMA) (500 mg, 1 equiv., 2.52 mmol), aniline (1.5 equiv., 3.8 mmol) and imidazole (1.7 g, 10 equiv., 25 mmol) were taken in a pressure tube equipped with a magnetic stir bar. The tube was fitted with a Teflon screw cap under an argon flow. The reaction mixture was heated to 140 ^{o}C in silicone oil bath and allowed to stir for 4.5 h. Upon completion of the reaction, the reaction mixture was cooled to room temperature. The reaction mixture was diluted with DCM (75 mL) and this solution was washed with 2 M HCl (50 mL). The organic extract was dried with anhyd. Na_2SO_4, filtered and concentrated under reduced pressure. The resulting residue was purified by silica gel flash column chromatography eluting with EA:Hexane (1:4) to yield the desired product.

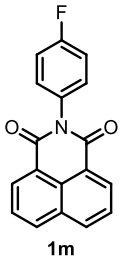
Analytical Data:


1k

2-Phenyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione;[2c] Yield: 72% (496 mg); Physical appearance: yellow solid; TLC R_f 0.30 (5:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.68 (d, J = 7.3 Hz, 2H), 8.30 (d, J = 8.3 Hz, 2H), 7.82 (t, J = 7.7 Hz, 2H), 7.63-7.55 (m, 2H), 7.54 – 7.48 (m, 1H), 7.35 (d, J = 7.4 Hz, 2H).

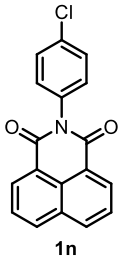

1l

2-(4-(*tert*-Butyl)phenyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione;[2c] Yield: 65% (560 mg); Physical appearance: brown solid; TLC R_f 0.45 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.67 (d, J = 7.2 Hz, 2H), 8.29 (d, J = 8.2 Hz, 2H), 7.82 (t, J = 7.7 Hz, 2H), 7.59 (d, J = 8.5 Hz, 2H), 7.27 (d, J = 8.2 Hz, 2H), 1.41 (s, 9H).



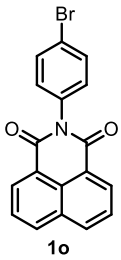
1m

2-(4-Fluorophenyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione;[2c] Yield: 72% (530 mg); Physical appearance: bright yellow solid; TLC R_f 0.25 (1:1, Petroleum ether: EA); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.68 (d, *J* = 7.3 Hz, 2H), 8.31 (d, *J* = 8.3 Hz, 2H), 7.83 (t, *J* = 8.0 Hz, 2H), 7.36 – 7.30 (m, 2H), 7.28 – 7.22 (m, 2H).



1n

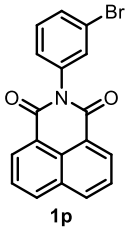
2-(4-Chlorophenyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione;[2c] Yield: 75% (581 mg); Physical appearance: bright yellow solid; TLC R_f 0.25 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.68 (d, *J* = 7.3 Hz, 2H), 8.31 (d, *J* = 8.2 Hz, 2H), 7.83 (t, *J* = 7.8 Hz, 2H), 7.55 (d, *J* = 8.5 Hz, 2H), 7.31 – 7.28 (m, 2H).



1o

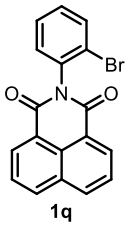
2-(4-Bromophenyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione:[2c] Yield: 81% (720 mg); Physical appearance: yellow solid; TLC R_f 0.35 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.68 (d, *J* = 7.2 Hz, 2H), 8.31 (d, *J* = 8.2 Hz, 2H), 7.83 (t, *J* = 7.7 Hz, 2H), 7.70 (d, *J* = 8.6 Hz, 2H), 7.23 (d, *J* = 8.6 Hz, 2H).

2-(3-Bromophenyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione;[2c] Yield: 52% (465 mg);



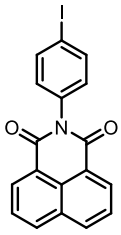
1p

Physical appearance: yellow solid; TLC R_f 0.40 (1:1, Petroleum ether: EA); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.67 (d, *J* = 7.3 Hz, 2H), 8.31 (d, *J* = 8.2 Hz, 2H), 7.83 (t, *J* = 7.8 Hz, 2H), 7.65 (d, *J* = 8.7 Hz, 1H), 7.58 – 7.50 (m, 1H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.33 – 7.29 (m, 1H).



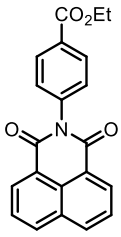
1q

2-(2-Bromophenyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione;[2c] **Yield**: 53% (467 mg); Physical appearance: Yellow solid; TLC R_f 0.50 (1:1, Petroleum ether: EA); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.70 (d, *J* = 7.2 Hz, 2H), 8.33 (d, *J* = 8.3 Hz, 2H), 7.91 – 7.76 (m, 3H), 7.60-7.49 (m, 1H), 7.47 – 7.36 (m, 2H).



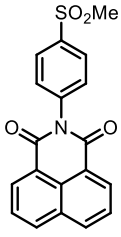
1r

2-(4-Iodophenyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione;[2c] Yield: 38% (380 mg); Physical appearance: yellow solid; TLC R_f 0.45 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.67 (d, *J* = 7.3 Hz, 2H), 8.31 (d, *J* = 8.4 Hz, 2H), 7.90 (d, *J* = 8.5 Hz, 2H), 7.82 (t, *J* = 7.8 Hz, 2H), 7.10 (d, *J* = 8.3 Hz, 2H).



1s

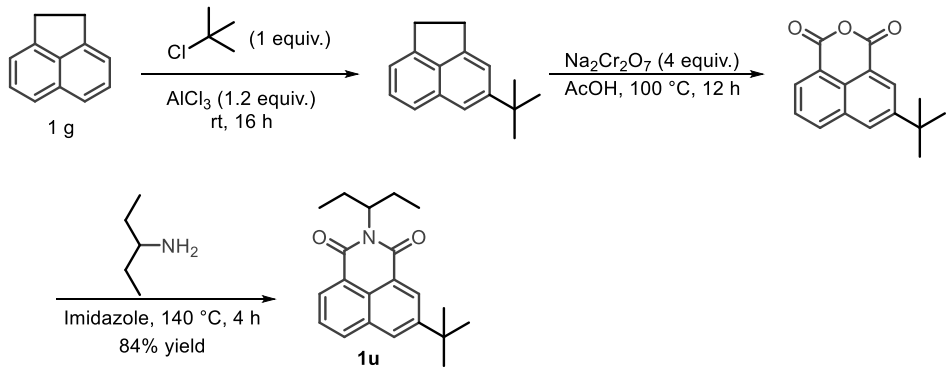
Ethyl 4-(1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)benzoate;[2c] Yield: 53% (465 mg); Physical appearance: white solid; TLC R_f 0.28 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.68 (d, *J* = 7.3 Hz, 2H), 8.32 (d, *J* = 8.3 Hz, 2H), 8.26 (d, *J* = 8.3 Hz, 2H), 7.83 (t, *J* = 7.8 Hz, 2H), 7.44 (d, *J* = 8.3 Hz, 2H), 4.45 (q, *J* = 7.1 Hz, 2H), 1.44 (t, *J* = 7.1 Hz, 3H).



1t

2-(4-(Methylsulfonyl)phenyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione;[2c] Yield: 60% (535 mg); Physical appearance: White solid; TLC R_f 0.20 (1:1, Petroleum ether: EA); **^{1}H NMR** (400 MHz, CDCl3) δ 8.69 (d, *J* = 7.3 Hz, 2H), 8.34 (d, *J* = 8.2 Hz, 2H), 8.16 (d, *J* = 8.4 Hz, 2H), 7.85 (t, *J* = 7.8 Hz, 2H), 7.59 (d, *J* = 8.4 Hz, 2H), 3.16 (s, 3H).

Scheme S5: Synthesis of *bay-tert*-butyl naphthalene monoimide (1u).[5]



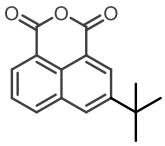
General Procedure for the synthesis of bay-tert-butyl naphthalene monoimide: Acenaphthene (1 g, 1 equiv., 6.5 mmol) and tertbutyl chloride (0.7 mL, 1 equiv., 6.5 mmol) were taken in an oven dried 50 mL round bottom flask. Then anhydrous aluminium trichloride (1.040 g, 1.2 equiv., 7.8 mmol) was added to the reaction mixture portion-wise during 30 minutes at 0 ^{o}C temperature. After addition of aluminium trichloride, the reaction was stirred at room temperature for 16 h.

After completion of the reaction, the reaction was quenched with ice-cold water and the organic layer was separated, and the aqueous phase was extracted with DCM (75 mL). The combined organic extract was dried over Na_2SO_4, filtered and concentrated in vacuum. The obtained white solid was used for the next step without further purification.

The prepared 3-*tert*-butyl acenaphthene (100 mg, 1 equiv., 0.47 mmol) was taken in an oven dried round bottom flask and dissolved in glacial acetic acid (2 mL). Then sodium dichromate (493 mg, 4 equiv., 2 mmol) was added to the reaction mixture and reflux the reaction mixture at 100 ^oC temperature for 12 h. After completion of the reaction, the reaction mixture was filtered through the Buchner funnel and washed the obtained green solid with water several timed until it becomes yellow solid. The obtained yellow solid was dried properly and purified through column chromatography by using Hexane:Ethyl acetate (4:1) as eluent.

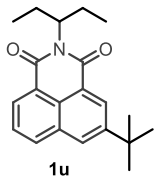
The obtained anhydride (50 mg, 1 equiv., 0.2 mmol) and imidazole (136 mg, 10 equiv., 2.0 mmol) were taken in an oven dried sealed tube equipped with a magnetic stir bar and then 3-amino pentane (24 μL, 1 equiv., 0.2 mmol) was added to the reaction mixture The tube was fitted with a Teflon screw cap under an argon flow. The reaction mixture was heated to 140 ^oC in silicone oil bath and allowed to stir for 4 h. Upon completion of the reaction, the reaction mixture was cooled to room temperature. The reaction mixture was diluted with DCM (75 mL) and this solution was washed with 2 M HCl (50 mL). The organic extract was dried with anhyd. Na_2SO_4, filtered and concentrated under reduced pressure. The resulting residue was purified by silica gel flash column chromatography eluting with EA:Hexane (1:9) to yield the desired product.

Analytical Data:



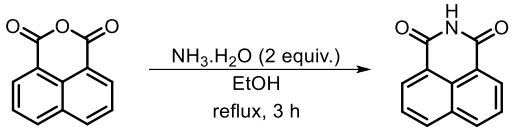
5-(*tert*-butyl)-1*H*,3*H*-benzo[*de*]isochromene-1,3-dione;[5] Yield: 48% (57 mg); Physical appearance: light yellow solid; TLC R_f 0.25 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.77 (s, 1H), 8.59 (d, J = 7.3 Hz, 1H), 8.31 (d, J = 8.2 Hz, 1H), 8.27 (s, 1H), 7.82 (t, J = 7.8 Hz, 1H), 1.52 (s, 9H).

5-(tert-butyl)-2-(pentan-3-yl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione;[5] Yield: 84% (54 mg);



Physical appearance: light yellow solid; TLC R_f 0.35 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.72 (s, 1H), 8.51 (d, *J* = 7.2 Hz, 1H), 8.21 – 8.11 (m, 2H), 7.77-7.68 (m, 1H), 5.14 – 5.03 (m, 1H), 2.35-2.20 (m, 2H), 2.00-1.83 (m, 2H), 1.49 (s, 9H), 0.92 (t, *J* = 7.5 Hz, 6H).

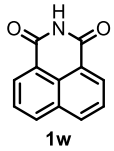
Scheme S5: Synthesis of 1,8-naphthalimide (1w).[6]



(1 equiv.) **1w** (98% yield)

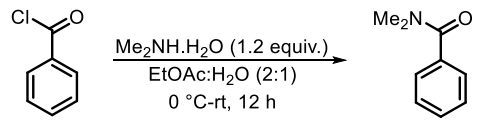
General Procedure for the synthesis of 1,8-naphthalimide: Naphthalene monoanhydride (NMA) (1 g, 1 equiv., 5 mmol) and ethanol (15 mL) were taken in an oven dried 50 mL round bottom flask, fitted with a reflux condenser. After that 25% conc. NH_3 (5 mL, 5 mmol, 2 equiv.) was added dropwise into the mixture, and the mixture was refluxed for 3 h in a paraffin oil bath. After completion of the reaction, the reaction mixture was cooled to room temperature and filtered. The resulting solid 1,8-naphthalimide was utilized for the next step without further purification.

Analytical Data:



1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione;[6] Yield: 98% (975 mg); Physical appearance: off-white solid; R_f 0.10 (DCM:Hex, 1:1); **^{1}H NMR (**500 MHz, $CDCl_3$) δ 8.63 (d, *J* = 7.3 Hz, 2H), 8.48 (br s, 1H), 8.29 (d, *J* = 8.1 Hz, 2H), 7.81 (t, *J* = 7.7 Hz, 2H).

Scheme S6: Synthesis of *N,N*-dimethyl benzamide (1y).[7]

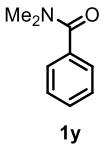


(1 equiv.) **1y** (98% yield)

General Procedure for the synthesis of N,N-dimethyl benzamide: To a suspension of potassium carbonate (2.37 g, 2 equiv., 17.2 mmol) in a 2:1 mixture of ethyl acetate (18 mL) and water (9

mL) was added dimethyl amine (1.8 mL, 1.2 equiv., 10.3 mmol). The resulting mixture was cooled to 0 °C, followed by dropwise addition of acid chloride (1mL, 1 equiv., 8.6 mmol) as a solution in ethyl acetate (5 mL). The biphasic mixture was warmed to room temperature and stirred for 12 h. The organic layer was separated, and the aqueous phase was extracted with EtOAc (75 mL). The combined organic extract was dried over Na_2SO_4, filtered and concentrated in vacuum.

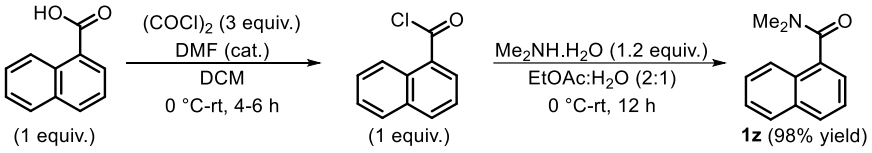
Analytical Data:



1y

***N,N*-dimethylbenzamide**;[7] Yield: 98% (1.26 g); Physical appearance: colorless liquid; R_f 0.50 (EA:Hex, 1:9); **^{1}H NMR (**500 MHz, $CDCl_3$) δ 7.45–7.20 (m, 1H), 2.92 (d, *J* = 64.4 Hz, 1H).

Scheme S7: Synthesis of *N,N*-dimethyl naphthalene-1-amide (1z).[8]

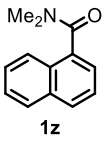


(1 equiv.) (1 equiv.) **1z** (98% yield)

General Procedure for the synthesis of N,N-dimethyl naphthalene-1-amide: To an oven-dried round bottom flask charged with magnetic stir bar, were added the 1-naphthoic acid (1 g, 1 equiv., 5.8 mmol) DMF (1 drop) and DCM (17 mL) under nitrogen atmosphere. Oxalyl chloride (1.5 mL, 3 equiv., 17.4 mmol) was added dropwise into it in ice cold condition. The ice bath was removed, and the reaction was stirred for 6 h at room temperature. The solution was removed under reduced pressure.

To a suspension of potassium carbonate (1.45 g, 2 equiv., 10.5 mmol) in a 2:1 mixture of ethyl acetate (10 mL) and water (5 mL) was added dimethyl amine (0.2 mL, 1.2 equiv., 6.3 mmol). The resulting mixture was cooled to 0 °C, followed by dropwise addition of acid chloride (1mL, 1 equiv., 5.2 mmol) as a solution in ethyl acetate (5 mL). The biphasic mixture was warmed to room temperature and stirred for 12 h. The organic layer was separated, and the aqueous phase was extracted with EtOAc (75 mL). The combined organic extract was dried over Na_2SO_4, filtered and concentrated in vacuum.

Analytical Data:



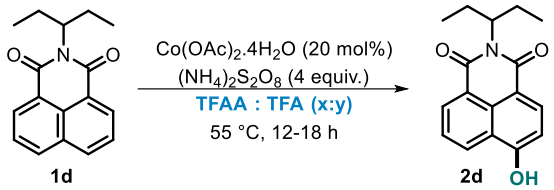
1z

***N,N*-dimethyl-1-naphthamide**;[8] Yield: 97% (1 g); Physical appearance: white solid; R_f 0.25 (EA:Hex, 1:4); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.94 – 7.85 (m, 2H), 7.85 – 7.78 (m, 1H), 7.59 – 7.48 (m, 3H), 7.47 – 7.41 (m, 1H), 3.28 (s, 3H), 2.83 (s, 3H).

2.1. Optimization studies:

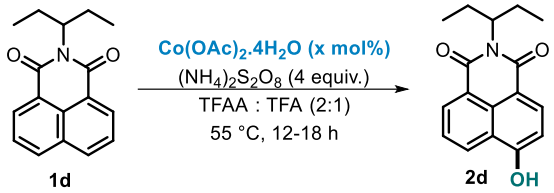
We began our optimization studies to obtain the best yield for this transformation. At first, we started screening the relative ratios of trifluoroacetic anhydride (TFAA) and trifluoroacetic acid (TFA) (**Table 1**). Excess amounts of TFAA resulted in the decomposition of the starting material. By varying the different ratios, it was revealed that the 2:1 ratio of TFAA and TFA is the optimum ratio in which a yield of 70% was obtained for the 4-hydroxy NMI (**Table 1**, **entry 4**). In the absence of TFAA, only a trace conversion of the NMI was observed, and without TFA, the reaction did not proceed (**entries 5 and 6**). These optimization studies indicated that the 2:1 ratio of TFAA and TFA must be maintained to obtain the *peri*-hydroxy-1,8-naphthalimide derivatives with good yield. The combination of acetic anhydride and acetic acid in a 2:1 ratio was found to be ineffective in facilitating the transformation of NMI into 4-hydroxy NMI (**entry 7**, **Table 1**). Increasing the stoichiometry of $Co(OAc)_2.4H_2O$ enhanced the reaction yield up to 82% (**Table 2** & **3**). Throughout the optimization studies, 82% was the highest yield obtained for this *peri*-C–H hydroxylation reaction. No drastic change was observed by lowering the stoichiometry of the oxidant (**Table 4**). Instead of ammonium persulphate, when other oxidants such as potassium persulphate or oxone were used, the reaction efficiency decreased, (**Table 5**). To check whether the source of the hydroxyl group is the acetate from the catalyst $Co(OAc)_2.4H_2O$, we carried out the reaction with other cobalt catalysts, such as $CoCl_2.6H_2O$, $CoBr_2$, $CoCl_2$ (**Table 6**, **entries 4-6**). All the three control reactions worked with the formation of 4-hydroxy-1,8-naphthalimides, albeit with lower reaction efficiency, which indicated that the hydroxy group did not come from the catalyst.

Table S1: Optimization of TFAA:TFA ratios



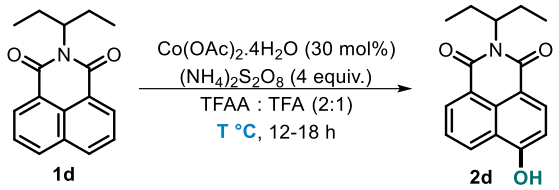
Entry	TFAA:TFA (x:y)	Yield
1	TFAA:TFA (9:1)	Complex reaction mixture
2	TFAA:TFA (1:1)	60%
3	TFAA:TFA (1:2)	51%
4	TFAA:TFA (2:1)	70%
5	TFA	trace
6	TFAA:DCE (2:1)	No reaction
7	Ac_2O:AcOH (2:1)	No reaction

Table S2: Optimization of the stoichiometry of catalyst



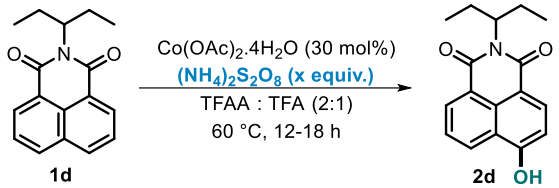
Entry	Catalyst (x mol%)	Yield
1	$Co(OAc)_2.4H_2O$ (20 mol%)	70%
2	$Co(OAc)_2.4H_2O$ (30 mol%)	75%
3	$Co(OAc)_2.4H_2O$ (10 mol%)	40%
4	$Co(OAc)_2.4H_2O$ (50 mol%)	33%
5	$Co(OAc)_2.4H_2O$ (100 mol%)	13%

Table S3: Optimization of temperature



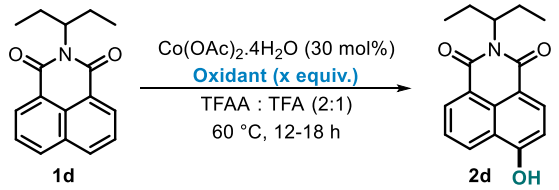
Entry	Temperature (°C)	Yield
1	55 °C	75%
2	60 °C	82 %
3	70 °C	Complex reaction mixture
4	40 °C	30%
5	25 °C	20%

Table S4: Optimization of the stoichiometry of the oxidant



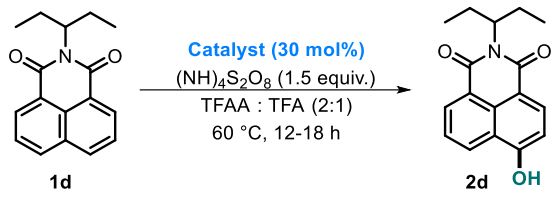
Entry	Oxidant (x equiv.)	Yield
1	$(NH_4)_2S_2O_8$ (4 equiv.)	82%
2	$(NH_4)_2S_2O_8$ (3 equiv.)	81%
3	$(NH_4)_2S_2O_8$ (2 equiv.)	82%
4	$(NH_4)_2S_2O_8$ (1.5 equiv.)	82%
5	$(NH_4)_2S_2O_8$ (10 equiv.)	71%

Table S5: Optimization of the oxidant



Entry	Oxidants	Yield
1	$(NH_4)_2S_2O_8$ (1.5 equiv.)	82%
2	$K_2S_2O_8$ (1.5 equiv.)	44%
3	Oxone (1.5 equiv.)	20%

Table S6: Optimization of the catalyst

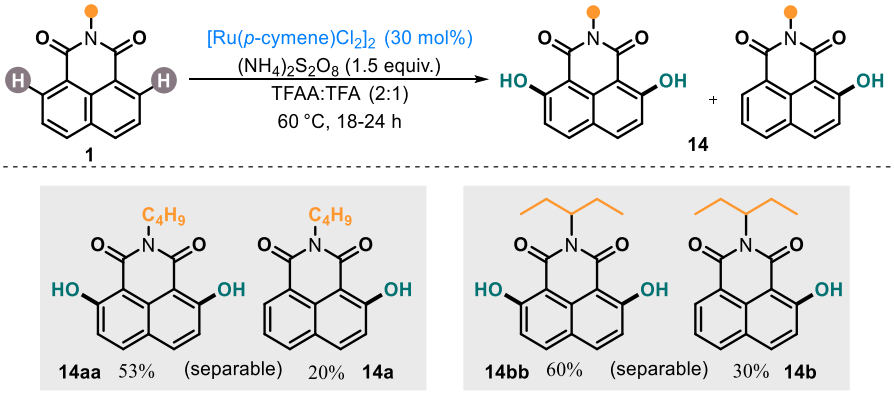


Entry	Catalysts	Yield of **2d**
1	$Co(OAc)_2.4H_2O$ (30 mol%)	82%
2	$Mn(OAc)_2$ (30 mol%)	19%
3	without catalyst	0%
4	$Co(OAc)_2$ (30 mol%)	80%
5	$CoCl_2.6H_2O$ (30 mol%)	38%
*6	$CoBr_2$ (30 mol%)	44%
7	$CoCl_2$ (30 mol%)	62%
8	$Cu(OAc)_2.H_2O$ (30 mol%)	75%
9	$Cu(OAc)_2$ (30 mol%)	62%
10	$Cu(OTf)_2$ (30 mol%)	trace
[a]11	$[Ru(p\text{-cymene})Cl_2]_2$	0%
[b]12	$Co(NO_3)_2.6H_2O$	trace

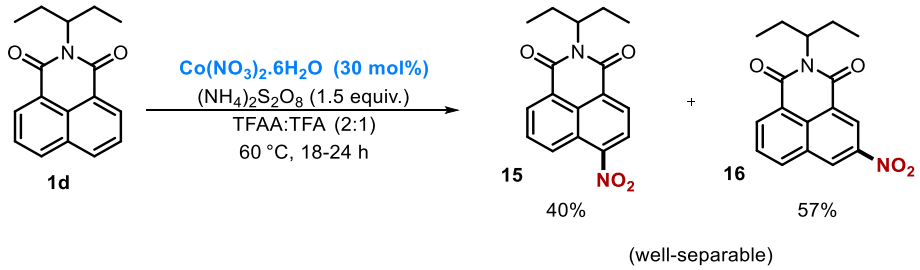
*Reaction time 36 h. [a]*ortho*-C–H hydroxylation of NMI, [b]mixture of *bay*- and *peri*-nitrated NMI

As expected, the reaction with a 4*d*-transition metal catalyst $[Ru(p\text{-cymene})Cl_2]_2$ energetically favoured the *ortho*-C–H bond activation pathway over the *peri*-C–H hydroxylation process. In this case, two well-separable mono- and di-substituted *ortho*-hydroxyl NMIs were obtained. The formation of the *ortho*-hydroxyl derivative was confirmed by the H-bonded *ortho*-OH proton's signal in the ^1H NMR spectra at δ ~13.5 ppm in the $CDCl_3$. We checked the Ru(II)-catalyzed reaction conditions with three different NMIs, and in each case, we observed the formation of corresponding *ortho*-hydroxylated derivatives (**Scheme S8**). The reaction with the catalyst $Co(NO_3)_2.6H_2O$ resulted in the formation of 4-hydroxy NMI in a very trace amount with the complete consumption of starting materials. The rest of the conversion resulted in the formation of *bay*- and *peri*-nitrated NMIs in major amounts (**Scheme S9**).

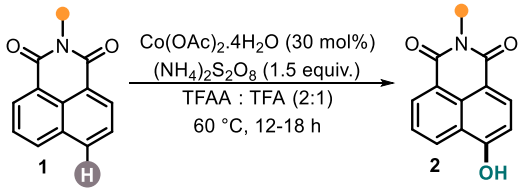
Scheme S8: Ru(II)-catalyzed *ortho*-C–H hydroxylation of NMI.



Scheme S9: Co(II)-catalyzed *bay*- and *peri*-nitration of NMI.



Scheme S10: General procedure for the synthesis of *peri*-hydroxy-*N*-substituted 1,8-naphthalimides (2).

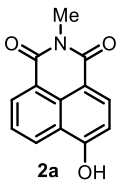


In an oven-dried pressure tube equipped with a magnetic stir bar, was placed *N*-substituted naphthalene monoimide 1 (0.1 mmol, 1 equiv.), cobalt diacetate tetrahydrate (7 mg, 0.03 mmol, 0.3 equiv.) and ammonium persulfate (34 mg, 0.15 mmol, 1.5 equiv.). Trifluoroacetic anhydride (0.2 mL) and trifluoroacetic acid (0.1 mL) with a volume ratio 2:1 were added. The tube was fitted with a Teflon screw cap and stirred at 60 ^{o}C in a paraffin oil bath for 12-18 h. After TLC analysis indicated the completion of the reaction, the reaction mixture was cooled to room temperature,

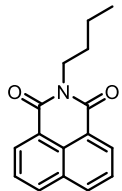
diluted with ethyl acetate (75 mL). The trifluoroacetic acid was quenched with aqueous solution of saturated sodium bicarbonate (25 mL). The organic layer was washed with water and dried over anhyd. Na_2SO_4. The extract was concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:4) or (1:1) as eluent to get the desired *peri*-hydroxylated NMIs.

Analytical Data:

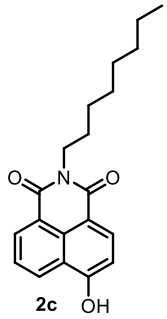
6-Hydroxy-2-methyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 61% (14 mg); Physical appearance: yellow solid; R_f 0.15 (1:4 EtOAc:Hex); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 11.87 (s, 1H), 8.49 (d, *J* = 8.0 Hz, 1H), 8.41 (d, *J* = 7.1 Hz, 1H), 8.30 (d, *J* = 8.0 Hz, 1H), 7.72 (t, *J* = 7.7 Hz, 1H), 7.12 (d, *J* = 8.0 Hz, 1H), 3.37 (s, 3H); **^{13}C{^{1}H} NMR** (176 MHz, DMSO-d_6) δ 164.4, 163.7, 160.7, 133.9, 131.5, 129.5, 129.3, 126.0, 122.8, 122.2, 113.1, 110.4, 26.9; **IR** (thin film, neat, cm^{-1}) 3308, 1691, 1652, 1580, 1361, 1297, 1268, 1024, 779, 756; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{13}H_8NO_3^-$ 226.0499, found 226.0477.



2-Butyl-6-hydroxy-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 65% (17 mg); Physical appearance: yellow solid; R_f 0.2 (4:1 Petroleum Ether:Ethylacetate); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 11.83 (br s, 1H), 8.57 – 8.37 (m, 2H), 8.35 – 8.28 (m, 1H), 7.95 – 7.57 (m, 1H), 7.22 – 7.04 (m, 1H), 4.06 – 3.93 (m, 2H), 1.59 (q, *J* = 8.1, 7.7 Hz, 2H), 1.39 – 1.28 (m, 2H), 0.92 (t, *J* = 6.5Hz, 3H); **^{13}C{^{1}H} NMR** (126 MHz, DMSO-d_6) δ 164.1, 163.4, 160.7, 133.9, 131.5, 129.6, 129.3, 125.9, 122.8, 122.2, 113.0, 110.4, 30.2, 20.3, 14.2; **IR** (thin film, neat, cm^{-1}) 3440, 2978, 2251, 2126, 1653, 1359, 1248, 1052, 1025, 1006, 747; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{16}H_{14}NO_3^-$ 268.0968, found 268.0952.

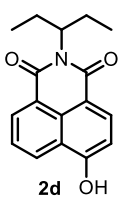


6-Hydroxy-2-octyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 67% (22 mg); Physical appearance: yellow solid; R_f 0.25 (4:1 Petroleum Ether:Ethylacetate); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.52 (d, *J* = 8.2 Hz, 1H), 8.45 (d, *J* = 7.0 Hz, 1H), 8.34 (d, *J* = 8.2 Hz, 1H), 7.74 (t, *J* = 7.8 Hz, 1H), 7.14 (d, *J* = 8.2 Hz, 1H), 3.99 (t, *J* = 7.3 Hz, 2H), 1.67 – 1.50 (m, 2H), 1.36 – 1.19 (m, 10H), 0.83 (t, *J* = 6.3 Hz, 3H); **^{13}C{^{1}H} NMR** (126 MHz, DMSO-d_6) δ 164.1, 163.4, 160.7, 133.9, 131.5, 129.6, 129.3, 126.0, 122.8, 122.3, 113.1, 110.4, 31.7, 29.2, 29.0, 28.0, 27.0, 22.5, 14.4; **IR** (thin film, neat, cm^{-}

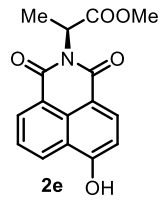


[1]) 3324, 2942, 2832, 1661, 1449, 1449, 1113, 1022, 627; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{20}H_{22}NO_3^-$ 324.1594, found 324.1618.

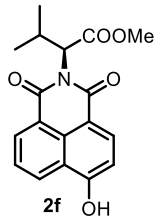
6-hydroxy-2-(pentan-3-yl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 82% (23 mg); Physical appearance: yellow solid; R_f 0.25 (Petroleum Ether:Ethylacetate, 4:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.69 – 8.57 (m, 2H), 8.48 (d, *J* = 7.8 Hz, 1H), 7.74 (t, *J* = 8.0 Hz, 1H), 7.12 (d, *J* = 8.1 Hz, 1H), 5.20 – 5.01 (m, 1H), 2.37 – 2.18 (m, 2H), 2.04 – 1.88 (m, 2H), 0.94 (t, *J* = 7.4 Hz, 6H); **^{13}C{^{1}H} NMR** (126 MHz, $CDCl_3$) δ 158.7, 130.1, 128.8, 125.9, 122.4, 110.2, 57.4, 25.1, 11.3; **IR** (thin film, neat, cm^{-1}) 3241, 2967, 1695, 1646, 1584, 1355, 1396, 1240, 1085, 1088, 783; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{17}H_{16}NO_3^-$ 282.1125, found 282.1133.



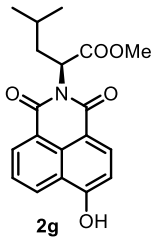
Methyl-*(S)*-2-(6-hydroxy-1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)propanoate; Yield: 80% (24 mg); Physical appearance: yellow solid; R_f 0.15 (1:4 EtOAc:Hex); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 12.05 (br s, 1H), 8.59 (d, *J* = 8.5 Hz, 1H), 8.52 (d, *J* = 7.5 Hz, 1H), 8.40 (d, *J* = 8.3 Hz, 1H), 7.80 (t, *J* = 7.9 Hz, 1H), 7.20 (d, *J* = 8.2, Hz, 1H), 5.67 (q, *J* = 7.0 Hz, 1H), 3.61 (s, 3H), 1.54 (d, *J* = 6.9 Hz, 3H); **^{13}C{^{1}H} NMR** (176 MHz, DMSO-d_6) δ 171.1, 163.6, 162.9, 161.3, 134.7, 132.2, 129.9, 129.8, 126.3, 122.9, 121.8, 112.5, 110.7, 52.5, 48.5, 15.0; **IR** (thin film, neat, cm^{-1}) 3326, 2944, 2832, 1450, 1021, 607; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{16}H_{12}NO_5^-$ 298.0710, found 298.0703; **$[\alpha]_D^{27.1}$** = –2.5 (*c* 0.1, MeOH).



Methyl-*(S)*-2-(6-hydroxy-1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)-3-methylbutanoate; Yield: 69% (22 mg); Physical appearance: yellow solid; R_f 0.15 (1:4 EtOAc:Hex); **^{1}H NMR (**500 MHz, $CDCl_3$) δ 8.98 (br s, 1H), 8.51 (d, *J* = 7.2 Hz, 1H), 8.28 (d, *J* = 8.3 Hz, 1H), 8.10 (d, *J* = 8.0 Hz, 1H), 7.53 (t, *J* = 7.8 Hz, 1H), 6.89 (d, *J* = 8.1 Hz, 1H), 5.48 (d, *J* = 9.0 Hz, 1H), 3.90 (s, 3H), 2.91–2.84 (m, 1H), 1.33 (d, *J* = 6.5 Hz, 3H), 0.80 (d, *J* = 6.9 Hz, 3H); **^{13}C{^{1}H} NMR** (126 MHz, $CDCl_3$) δ 172.9, 164.5, 164.0, 163.6, 159.2, 134.1, 132.0, 129.5, 129.3, 125.6, 122.2, 121.3, 113.4, 110.1, 58.8, 53.028, 27.5, 22.2, 19.1; **IR** (thin film, neat, cm^{-1}) 3302, 2927, 1696, 1659, 1587, 1457, 1376, 1282, 1242, 1214, 1113, 1074, 1021, 754, 668, 489; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{18}H_{16}NO_5^-$ 326.1023, found 326.1050; **$[\alpha]_D^{29.1}$** = + 216 (*c* 0.33, $CHCl_3$).

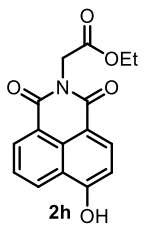


Methyl-*(S)*-2-(6-hydroxy-1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)-4-methylpentanoate; Yield: 79% (27 mg); Physical appearance: yellow solid; R_f : 0.1 (4:1 Hexane: Ethyl acetate); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.76 (br s, 1H), 8.49 (d, *J* = 7.0 Hz, 1H), 8.13 (d, *J* = 8.3 Hz, 1H), 8.05 (d, *J* = 8.1 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 1H), 6.81 (d, *J* = 8.1 Hz, 1H), 5.91 (dd, *J* = 9.1, 5.0 Hz, 1H), 3.95 (s, 3H), 2.27–2.19 (m, 1H), 2.18 – 2.11 (m, 1H), 1.59–1.48 (m, 1H), 1.02 (d, *J* = 6.5 Hz, 3H), 0.92 (d, *J* = 6.6 Hz, 3H); **^{13}C{^{1}H} NMR** (126 MHz, $CDCl_3$) δ 173.6, 164.4, 163.4, 159.0, 134.1, 131.8, 129.3, 129.2, 125.5, 122.1, 121.4, 113.6, 110.0, 53.3, 52.1, 38.1, 25.3, 23.2, 22.1; **IR** (thin film, neat, cm^{-1}) 3286, 2957, 1746, 1695, 1659, 1588, 1456, 1373, 1271, 1240, 1115, 1074, 1007, 757, 782; **ESI-HRMS:** $[M+H]^+$ Calculated for $C_{19}H_{20}NO_5^+$ 364.1155, found 364.1163; **$[\alpha]_D^{29}$** = + 202 (*c* 0.33, $CHCl_3$).



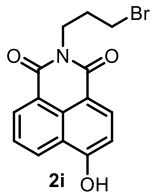
2g

Ethyl 2-(6-hydroxy-1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)acetate; Yield: 63% (19 mg); Physical appearance: solid; R_f 0.1 (4:1 Hexane: Ethyl acetate); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 12.00 (br s, 1H), 8.49-8.58 (m, 1H), 8.40-8.49 (m, 1H), 8.39 – 8.30 (m, 1H), 7.86 – 7.69 (m, 1H), 7.22 – 7.11 (m, 1H), 4.81 – 4.74 (m, 2H), 4.16 (q, *J* = 7.1, 2H), 1.43–1.25 (m, 3H); **^{13}C{^{1}H} NMR** (126 MHz, DMSO-d_6) δ 168.7, 163.827, 163.0, 161.4, 134.5, 131.9, 129.9, 129.7, 126.1, 122.9, 121.6, 112.3, 110.6, 61.5, 41.4, 14.5; **IR** (thin film, neat, cm^{-1}) 3432, 2979, 2251, 2126, 1660, 1279, 1245, 1053, 1006, 1025, 745; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{16}H_{12}NO_5^-$ 298.0696, found 298.0710.



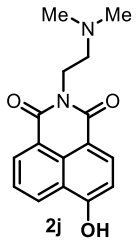
2h

2-(3-Bromopropyl)-6-hydroxy-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 70% (23 mg); Physical appearance: solid; R_f 0.30 (4:1 Hexane: Ethyl acetate); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 11.86 (s, 1H), 8.52 (d, *J* = 8.3 Hz, 1H), 8.45 (d, *J* = 7.3 Hz, 1H), 8.34 (d, *J* = 8.2 Hz, 1H), 7.75 (t, *J* = 7.8 Hz, 1H), 7.14 (d, *J* = 8.2 Hz, 1H), 4.14 (t, *J* = 6.9 Hz, 2H), 3.60 (t, *J* = 6.6 Hz, 2H), 2.25 – 2.13 (m, 2H); **^{13}C{^{1}H} NMR** (176 MHz, DMSO-d_6) δ 164.3, 163.6, 160.7, 134.0, 131.6, 129.7, 129.4, 126.0, 122.8, 122.3, 113.1, 110.4, 38.9, 32.8, 31.5; **IR** (thin film, neat, cm^{-1}) 3392, 3012, 2351, 2231, 1750, 1680, 1267, 1132, 1011, 1022, 745; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{15}H_{11}BrNO_3^-$ 331.9917 and 333.9897, found 331.9939 and 333.9916.



2i

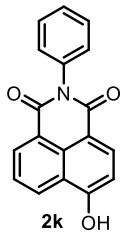
2-(2-(Dimethylamino)ethyl)-6-hydroxy-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 56% (16 mg); Physical appearance: Orange solid; R_f 0.15 (1:1 Methanol: Ethyl acetate);



2j

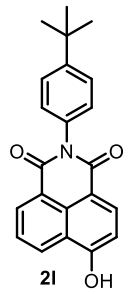
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.39 (d, J = 7.9 Hz, 1H), 8.22 (d, J = 7.3 Hz, 1H), 7.96 (d, J = 8.9 Hz, 1H), 7.28 (t, J = 7.6 Hz, 1H), 6.21 (d, J = 8.9 Hz, 1H), 4.19 – 4.05 (m, 2H), 2.45 (t, J = 7.1 Hz, 2H), 2.21 (s, 6H); **^{13}C{^{1}H} NMR** (176 MHz, DMSO-d_6) δ 177.7, 164.9, 162.8, 135.8, 132.9, 131.5, 130.3, 128.3, 121.0, 120.9, 114.6, 98.6, 57.4, 45.9, 37.2; **IR** (thin film, neat, cm^{-1}) 3431, 2830, 2362, 2231, 1653, 1281, 1223, 1068, 987, 951, 748; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{16}H_{15}N_2O_3^-$ 283.1077, found 283.1067.

6-Hydroxy-2-phenyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 68% (20 mg); Physical appearance: pale yellow solid; TLC R_f 0.25 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 11.92 (br s, 1H), 8.61 (d, J = 8.3 Hz, 1H), 8.49 (d, J = 7.2 Hz, 1H), 8.37 (d, J = 8.2 Hz, 1H), 7.80 (t, J = 7.8 Hz, 1H), 7.52 (t, J = 7.5 Hz, 2H), 7.45 (t, J = 7.4 Hz, 1H), 7.35 (d, J = 7.5 Hz, 2H), 7.19 (d, J = 8.1 Hz, 1H); **^{13}C{^{1}H} NMR** (176 MHz, DMSO-d_6) δ 164.4, 163.7, 136.8, 134.1, 131.7, 130.2, 129.6, 129.6, 129.2, 128.4, 126.1, 123.1, 122.8, 110.5; **IR** (thin film, neat, cm^{-1}) 3442, 2988, 2261, 2134, 1637, 1359, 1228, 1012, 1022, 1001, 748; **ESI-HRMS**: Calculated for $C_{18}H_{12}NO_3^+$ $[M+H]^+$ 290.0812, found 290.0806.



2k

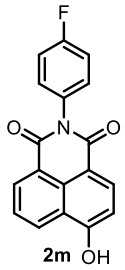
2-(4-(*tert*-Butyl)phenyl)-6-hydroxy-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 61% (21 mg); Physical appearance: pale yellow solid; TLC R_f 0.30 (1:1, Petroleum ether: EA);



2l

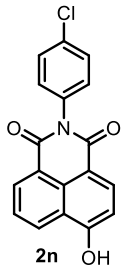
^{1}H NMR (500 MHz, DMSO-d_6) δ 11.95 (br s, 1H), 8.63-8.54 (m, 1H), 8.52-8.44 (m, 1H), 8.40-8.33 (m, 1H), 7.87-7.73 (m, 1H), 7.52 (d, J = 8.3 Hz, 2H), 7.25 (d, J = 8.1 Hz, 2H), 7.22-7.15 (m, 1H), 1.36 (s, 9H); **^{13}C{^{1}H} NMR** (126 MHz, DMSO-d_6) δ 164.5, 163.8, 160.8, 150.7, 134.1, 131.7, 131.7, 130.1, 129.5, 129.1, 126.1, 126.1, 122.9, 122.7, 113.4, 110.4, 34.9, 31.7; **IR** (thin film, neat, cm^{-1}) 3540, 2968, 2241, 2123, 1658, 1367, 1244, 1032, 1225, 1087, 687; **ESI-HRMS**: Calculated for $C_{22}H_{19}NO_3Na^+$ $[M+Na]^+$ 368.1257, found 368.1256.

2-(4-Fluorophenyl)-6-hydroxy-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 82% (26 mg);



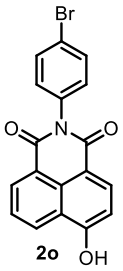
Physical appearance: pale yellow solid; TLC R_f 0.25 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 11.95 (br s, 1H), 8.60 (d, J = 8.4 Hz, 1H), 8.49 (d, J = 7.3 Hz, 1H), 8.37 (d, J = 8.1 Hz, 1H), 7.81 (t, J = 7.8 Hz, 1H), 7.49-7.39 (m, 2H), 7.39-7.29 (m, 2H), 7.20 (d, J = 8.0 Hz, 1H); **^{13}C{^{1}H} NMR** (126 MHz, DMSO-d_6) δ 164.5, 163.8, 162.0 ($J_{C\text{-}F}$ = 244.8 Hz), 160.9, 134.1, 132.9 ($J_{C\text{-}F}$ = 3.0 Hz), 131.7 ($J_{C\text{-}F}$ = 8.9 Hz), 130.2, 129.6, 126.1, 123.0, 122.8, 116.1 ($J_{C\text{-}F}$ = 22.5 Hz), 113.4, 110.5; **^{19}F NMR** (471 MHz, DMSO-d_6) δ –114.45; **IR** (thin film, neat, cm^{-1}) 3840, 2979, 2261, 2143, 1634, 1320 1256, 1153, 1028, 989, 747; **ESI-HRMS**: Calculated for $C_{18}H_{11}FNO_3^+$ $[M+H]^+$ 308.0717, found 308.0716.

2-(4-Chlorophenyl)-6-hydroxy-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 86% (27 mg);



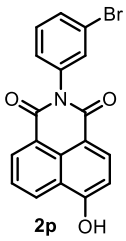
Physical appearance: pale yellow solid; TLC R_f 0.30 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 11.94 (br s, 1H), 8.66 – 8.56 (m, 1H), 8.52 – 8.42 (m, 1H), 8.41 – 8.30 (m, 1H), 7.85 – 7.73 (m, 1H), 7.58 (d, J = 8.7 Hz, 2H), 7.41 (d, J = 8.5 Hz, 2H), 7.19 (t, J = 8.3 Hz, 1H); **^{13}C{^{1}H} NMR** (126 MHz, DMSO-d_6) δ 164.3, 163.6, 161.1, 135.7, 134.1, 133.1, 131.7, 131.6, 130.2, 129.6, 129.3, 126.1, 123.0, 122.7, 113.3, 110.5; **IR** (thin film, neat, cm^{-1}) 3460, 2980, 2351, 2026, 1453, 13259, 1148, 1051, 925, 659; **ESI-HRMS**: Calculated for $C_{18}H_{11}ClNO_3^+$ $[M+H]^+$ 324.0422, found 324.0428.

2-(4-Bromophenyl)-6-hydroxy-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 80% (29 mg);



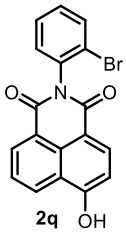
Physical appearance: pale yellow solid; TLC R_f 0.3 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 11.95 (br s, 1H), 8.66-8.54 (m, 1H), 8.54-8.42 (m, 1H), 8.42-8.30 (m, 1H), 7.84 – 7.76 (m, 1H), 7.72 (d, J = 8.2 Hz, 2H), 7.35 (d, J = 8.1 Hz, 2H), 7.19 (t, J = 7.6 Hz, 1H); **^{13}C{^{1}H} NMR** (126 MHz, DMSO-d_6) δ 164.3, 163.6, 161.1, 135.7, 134.1, 133.1, 131.7, 131.6, 130.2, 129.6, 129.3, 126.1, 123.0, 122.7, 113.3, 110.5; **IR** (thin film, neat, cm^{-1}) 3603, 2878, 2451, 2026, 1723, 1429, 1198, 1052, 1026, 747; **ESI-HRMS**: Calculated for $C_{18}H_{10}BrNO_3Na^+$ $[M+Na]^+$ 389.9763 and 391.9717, found 389.9750 and 391.9728.

2-(3-Bromophenyl)-6-hydroxy-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 65% (24 mg);



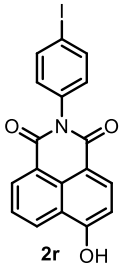
Physical appearance: pale yellow solid; TLC R_f 0.36 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 8.60 (d, J = 8.3 Hz, 1H), 8.47 (d, J = 7.2 Hz, 1H), 8.35 (d, J = 8.1 Hz, 1H), 7.78 (t, J = 7.8 Hz, 1H), 7.71 – 7.63 (m, 2H), 7.49 (t, J = 8.2 Hz, 1H), 7.40 (d, J = 7.9 Hz, 1H), 7.14 (d, J = 8.3 Hz, 1H); **^{13}C{^{1}H} NMR** (176 MHz, DMSO-d_6) δ 164.4, 163.6, 138.5, 134.3, 132.7, 131.6, 131.4, 131.1, 130.5, 129.8, 129.1, 125.8, 123.4, 122.6, 121.6, 110.7; **IR** (thin film, neat, cm^{-1}) 3659, 2878, 2351, 2026, 1853, 1559, 1348, 1019, 980; **ESI-HRMS**: Calculated for $C_{18}H_{10}BrNO_3Na^+$ $[M+Na]^+$ 389.9736 and 391.9717, found 389.9713 and 391.9687.

2-(2-Bromophenyl)-6-hydroxy-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 48% (17 mg);



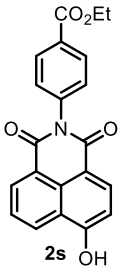
Physical appearance: pale yellow solid; TLC R_f 0.25 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 12.05 (br s, 1H), 8.64 (d, J = 8.4 Hz, 1H), 8.53 (d, J = 7.3 Hz, 1H), 8.42 (d, J = 8.2 Hz, 1H), 7.83 (d, J = 7.9 Hz, 2H), 7.56 (d, J = 4.6 Hz, 2H), 7.48 – 7.40 (m, 1H), 7.23 (d, J = 8.2 Hz, 1H); **^{13}C{^{1}H} NMR** (126 MHz, DMSO-d_6) δ 163.7, 162.9, 161.3, 136.2, 134.5, 133.1, 132.0, 131.8, 130.8, 130.3, 130.0, 129.0, 126.2, 123.2, 123.1, 122.3, 112.9, 110.6; **IR** (thin film, neat, cm^{-1}) 3823, 2975, 2051, 1654, 1360, 1348, 1048, 1002, 847; **ESI-HRMS**: Calculated for $C_{18}H_{10}BrNO_3Na^+$ $[M+Na]^+$ 389.9736 and 391.9717, found 389.9741 and 391.9727.

6-Hydroxy-2-(4-iodophenyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 74% (30 mg);

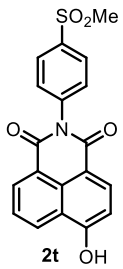


Physical appearance: pale yellow solid; TLC R_f 0.40 (9:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 11.96 (br s, 1H), 8.61 (d, J = 8.7 Hz, 1H), 8.49 (d, J = 7.2 Hz, 1H), 8.37 (d, J = 8.3 Hz, 1H), 7.88 (d, J = 8.3 Hz, 2H), 7.81 (t, J = 7.8 Hz, 1H), 7.19 (d, J = 8.4 Hz, 2H); **^{13}C{^{1}H} NMR** (176 MHz, DMSO-d_6) δ 164.3, 163.6, 160.9, 138.2, 136.7, 134.2, 132.1, 131.8, 130.2, 129.6, 126.1, 123.0, 122.7, 113.4, 110.4, 94.7; **IR** (thin film, neat, cm^{-1}) 3840, 2978, 2251, 2326, 1653, 1359, 1448, 1052, 1029, 1006, 737; **ESI-HRMS**: Calculated for $C_{18}H_{11}INO_3^+$ $[M+H]^+$ 415.9778, found 415.9773.

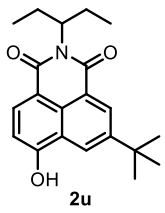
Ethyl 4-(6-hydroxy-1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)benzoate; Yield: 73% (26 mg); Physical appearance: pale yellow solid; TLC R_f 0.20 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 11.98 (br s, 1H), 8.59 (d, J = 8.6 Hz, 1H), 8.48 (d, J = 7.3 Hz, 1H), 8.36 (d, J = 8.0 Hz, 1H), 8.10 (d, J = 8.4 Hz, 2H), 7.79 (t, J = 7.8 Hz, 1H), 7.54 (d, J = 8.2 Hz, 2H), 7.19 (d, J = 7.9 Hz, 1H), 4.38 (q, J = 7.1 Hz, 2H), 1.37 (t, J = 7.1 Hz, 3H); **^{13}C{^{1}H} NMR** (126 MHz, DMSO-d_6) δ 165.8, 164.3, 163.5, 161.0, 141.2, 134.2, 131.7, 130.3, 130.2, 130.1, 129.7, 126.1, 123.0, 122.6, 113.3, 110.5, 61.4, 14.6; **IR** (thin film, neat, cm^{-1}) 3604, 2878, 2451, 2026, 1723, 1429, 1198, 1053, 1028, 747; **ESI-HRMS**: Calculated for $C_{21}H_{15}NO_5Na^+$ $[M+Na]^+$ 384.0842, found 384.0838.



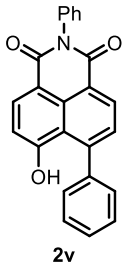
6-Hydroxy-2-(4-(methylsulfonyl)phenyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; **Yield**: 80% (29 mg); Physical appearance: pale yellow solid; TLC R_f 0.20 (3:7, Petroleum ether: EA); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 11.99 (br s, 1H), 8.66 – 8.54 (m, 1H), 8.54 – 8.42 (m, 1H), 8.38-8.33 (m, 1H), 8.09 (d, J = 5.6 Hz, 2H), 7.87 – 7.73 (m, 1H), 7.70 (d, J = 8.1 Hz, 2H), 7.26-7.12 (m, 1H), 3.34 (s, 3H); **^{13}C{^{1}H} NMR** (126 MHz, DMSO-d_6) δ 164.3, 163.6, 161.1, 141.6, 140.9, 134.2, 134.2, 131.8, 130.9, 130.3, 130.2, 129.7, 128.1, 126.1, 123.0, 122.6, 122.6, 113.2, 110.5, 43.8; IR (thin film, neat, cm-1) 3583, 2750, 2236, 1686, 1425, 1117, 998, 869; **ESI-HRMS**: Calculated for $C_{19}H_{14}NO_5S^+$ $[M+H]^+$ 368.0587, found 368.0589.



5-(tert-butyl)-7-hydroxy-2-(pentan-3-yl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; **Yield**: 42% (14 mg); Physical appearance: yellow solid; TLC R_f 0.25 (4:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 11.86 (s, 1H), 8.56 (s, 1H), 8.48 (d, J = 2.0 Hz, 1H), 8.30 (d, J = 8.2 Hz, 1H), 7.15 (d, J = 8.2 Hz, 1H), 4.99 – 4.87 (m, 1H), 2.23 – 2.08 (m, 2H), 1.87 – 1.74 (m, 2H), 1.44 (s, 9H) 0.79 (t, J = 7.4 Hz, 6H); **^{13}C{^{1}H} NMR** (176 MHz, DMSO-d_6) δ 160.4, 148.7, 128.2, 124.2, 122.7, 110.6, 79.6, 35.4, 31.43, 24.9, 11.5; **IR** (thin film, neat, cm-1) 3658, 2650, 2284, 1625, 1311, 1156, 959, 820, 780; **ESI-HRMS**: Calculated for $C_{21}H_{25}NO_3Na^+$ $[M+Na]^+$ 362.1727, found 362.1699.



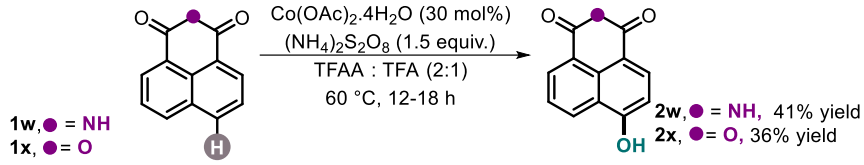
6-hydroxy-2,7-diphenyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; **Yield**: 39% (15 mg);



2v

Physical appearance: yellow solid; TLC R_f 0.25 (7:3, Petroleum ether: EA); **^{1}H NMR** (400 MHz, DMSO- d_6) δ 8.49 (d, J = 7.5 Hz, 1H), 8.41 (d, J = 8.1 Hz, 1H), 7.56 – 7.50 (m, 3H), 7.49 – 7.45 (m, 1H), 7.44 – 7.35 (m, 7H), 7.08 (d, J = 8.2 Hz, 1H); **^{13}C{^{1}H} NMR** (126 MHz, DMSO- d_6) δ 169.0, 168.6, 167.1, 151.5, 148.1, 141.4, 136.3, 134.3, 134.0, 133.6, 133.2, 132.3, 132.0, 126.7, 125.1, 118.2, 116.7, 99.9; **IR** (thin film, neat, cm-1) 3564, 2789, 1734, 1682, 1365, 1102, 908, 812, 788; **ESI-HRMS**: Calculated for $C_{24}H_{15}NO_3$ $[M+H]^+$ 366.1125, found 366.1117.

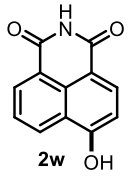
Scheme S11: General procedure for the synthesis of *peri*-hydroxy 1,8-naphthalimide (2w) and 1,8-naphthalic anhydride (2x).



In an oven-dried pressure tube equipped with a magnetic stir bar was placed 1,8-naphthalimide **1w** (0.05 mmol, 1 equiv.) or 1,8-naphthalic anhydride **1x** (0.05 mmol, 1 equiv.), cobalt diacetate tetrahydrate (4 mg, 0.015 mmol, 0.3 equiv.) and ammonium persulfate (17 mg, 0.075 mmol, 1.5 equiv.). Trifluoroacetic anhydride (0.4 mL) and trifluoroacetic acid (0.2 mL) with volume ratio 2:1 were added to this. The tube was fitted with a Teflon screw cap and the reaction mixture was stirred at 60 ^{o}C in a paraffin oil bath for 12–18 h. The reaction mixture was then cooled to room temperature, diluted with ethyl acetate (75 mL). The trifluoroacetic acid was quenched with an aqueous solution of saturated sodium bicarbonate (25 mL). After extraction, the organic layer was washed with water and dried over Na_2SO_4. This was concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:1) or (7:3) as eluent to get the desired *peri*-hydroxylated NMIs.

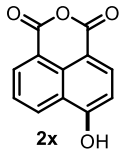
Analytical Data:

6-Hydroxy-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 41% (4 mg); Physical appearance:



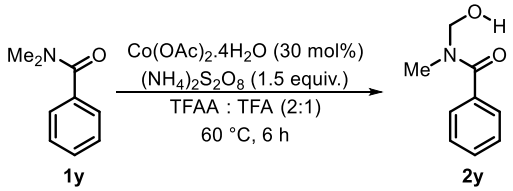
pale yellow solid; TLC R_f 0.10 (3:7, Petroleum ether: EA); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.45 (s, 1H), 8.55 (d, J = 8.0 Hz, 1H), 8.43 (d, J = 6.7 Hz, 1H), 8.31 (d, J = 7.9 Hz, 1H), 7.76 (t, J = 7.3 Hz, 1H), 7.14 (d, J = 7.9 Hz, 1H); **^{13}C{^{1}H} NMR** (176 MHz, DMSO-d_6) δ 164.9, 164.2, 133.2, 131.1, 130.9, 129.4, 125.9, 123.3, 122.8, 110.3; **IR** (thin film, neat, cm^{-1}) 3442, 2842, 1688, 1276, 1116, 1024, 751; **ESI-HRMS**: Calculated for $C_{12}H_8NO_3^+$ $[M+H]^+$ 214.0499, found 214.0488.

6-Hydroxy-1*H*,3*H*-benzo[*de*]isochromene-1,3-dione; Yield: 36% (4 mg); Physical appearance:



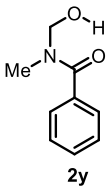
pale yellow solid; TLC R_f 0.10 (3:7, Petroleum ether: EA); **^{1}H NMR** (500 MHz, DMSO-d_6) δ 12.29 (s, 1H), 8.62 (d, J = 8.3 Hz, 1H), 8.52 (d, J = 7.2 Hz, 1H), 8.41 (d, J = 8.2 Hz, 1H), 7.82 (t, J = 7.8 Hz, 1H), 7.20 (d, J = 8.2 Hz, 1H); **^{13}C{^{1}H} NMR** (176 MHz, DMSO-d_6) δ 162.3, 161.9, 160.8, 136.2, 133.5, 132.4, 130.7, 126.4, 123.2, 119.0, 111.1; **IR** (thin film, neat, cm^{-1}) 3542, 2938, 1582, 1476, 1316, 1022, 668; **ESI-HRMS**: Calculated for $C_{12}H_7O_4^+$ $[M-H]^-$ 215.0339, found 215.0337.

Scheme S12: General procedure for the cobalt(II)-catalyzed C(*sp*^{3})-H methyl hydroxylation of *N*,*N*-dimethyl benzamide (1y).



In an oven-dried pressure tube equipped with a magnetic stir bar, was placed *N*,*N*-dimethyl benzamide **1y** (20 mg, 0.13 mmol, 1 equiv.), cobalt diacetate tetrahydrate (10 mg, 0.04 mmol, 0.3 equiv.) and ammonium persulfate (46 mg, 0.20 mmol, 1.5 equiv.). Trifluoroacetic anhydride (0.2 mL) and trifluoroacetic acid (0.1 mL) with a volume ratio 2:1 were added. The tube was fitted with a Teflon screw cap and stirred at 60 °C in a paraffin oil bath for 12-18 h. After TLC analysis indicated the completion of the reaction, the reaction mixture was cooled to room temperature, diluted with ethyl acetate (75 mL). The trifluoroacetic acid was quenched with aqueous solution of saturated sodium bicarbonate (25 mL). The organic layer was washed with water and dried over

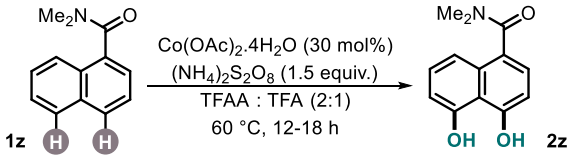
anhyd. Na_2SO_4. The extract was concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:4) as eluent to get the desired *N*-methyl-hydroxylated *N,N*-dimethyl benzamide.



2y

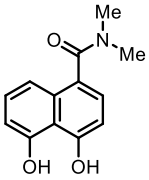
***N*-(Hydroxymethyl)-*N*-methylbenzamide**; Yield: 81% (17 mg); Physical appearance: pale yellow gel; TLC R_f 0.25 (4:1, Petroleum ether: EA); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.24 (s, 1H), 7.55 – 7.37 (m, 5H), 5.00 (d, *J* = 5.3 Hz, 2H), 3.12 (s, 3H); **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 173.34, 134.43, 130.62, 128.56, 127.25, 52.62, 38.18; **IR** (thin film, neat, cm^{-1}) 3649, 2170, 1470, 1425, 1321, 998, 668; **ESI-HRMS**: Calculated for molecular fragment after liberation of hydroxyl group from the molecule: $C_9H_{12}NO^+$ $[M+H]^+$ 150.0913, found 150.0907.

Scheme S13: General procedure for the cobalt(II)-catalyzed *peri*-C-H dihydroxylation *N,N*-dimethyl naphthalene-1-amide (2z).



In an oven-dried pressure tube equipped with a magnetic stir bar, was placed *N,N*-dimethyl naphthalene-1-amide 1 (30 mg, 0.15 mmol, 1 equiv.), cobalt diacetate tetrahydrate (11 mg, 0.04 mmol, 0.3 equiv.) and ammonium persulfate (51 mg, 0.2 mmol, 1.5 equiv.). Trifluoroacetic anhydride (0.4 mL) and trifluoroacetic acid (0.2 mL) with a volume ratio 2:1 were added. The tube was fitted with a Teflon screw cap and stirred at 60 ^{o}C in a paraffin oil bath for 12-18 h. Then the reaction mixture was cooled to room temperature, diluted with ethyl acetate (75 mL). The trifluoroacetic acid was quenched with aqueous solution of saturated sodium bicarbonate (25 mL). The organic layer was washed with water and dried over anhyd. Na_2SO_4. The extract was concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:1) as eluent to get the product.

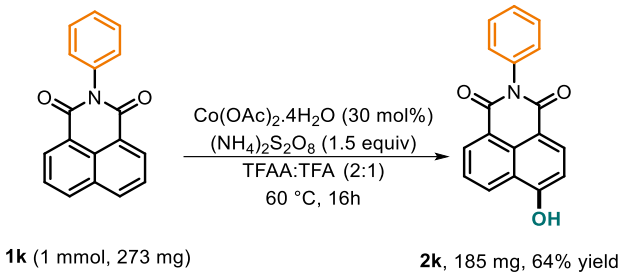
4,5-Dihydroxy-*N,N*-dimethyl-1-naphthamide; Yield: <5% (<1 mg); Physical appearance: pale yellow gel; TLC R_f 0.25 (4:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.17 (d, *J* = 7.7 Hz, 1H), 7.82 (t, *J* = 7.5 Hz, 1H), 7.60 (d, *J* = 7.6 Hz, 1H), 7.06-7.95 (m, 2H), 3.24 (s, 3H),



2z

3.01 (s, 3H).; **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) Due to very trace amount of reaction conversion, ^{13}C NMR of the compound **2v** could not be recorded; **IR** (thin film, neat, cm^{-1}) 3659, 2900, 1675, 1468, 1299, 1102, 668; **ESI-HRMS**: Calculated for $C_{13}H_{13}NO_3$ $[M-H]^-$ 230.0812, found 230.0835.

Scheme S14: Procedure for the *peri*-C–H hydroxylation of *N*-phenyl 1,8-naphthalimides (1k) on 1 mmol scale.

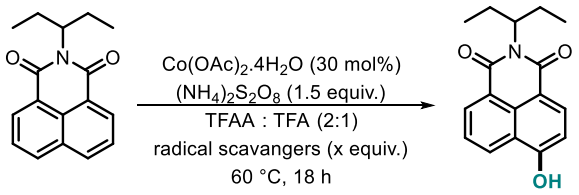


1k (1 mmol, 273 mg)

2k, 185 mg, 64% yield

In an oven-dried pressure tube equipped with a magnetic stir bar, was placed *N*-substituted naphthalene monoimide (1 mmol, 1 equiv.), cobalt diacetate tetrahydrate (74 mg, 0.3 mmol, 0.3 equiv.) and ammonium persulfate (342 mg, 1.5 mmol, 1.5 equiv.). Trifluoroacetic anhydride (2 mL) and trifluoroacetic acid (1 mL) with volume ratio 2:1 were added to this. The tube was fitted with a Teflon screw cap and the reaction mixture was stirred at 60 ^{o}C in a paraffin oil bath for 16 h. After TLC analysis indicated the completion of the reaction, the reaction mixture was cooled to room temperature, diluted with ethyl acetate (125 mL). The trifluoroacetic acid was quenched with an aqueous solution of saturated sodium bicarbonate (40 mL) and extracted. The organic layer was washed with water and dried over anhyd. Na_2SO_4. The solution was filtered and concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:4) or (1:1) as eluent to get the desired *peri*-hydroxylated NMIs.

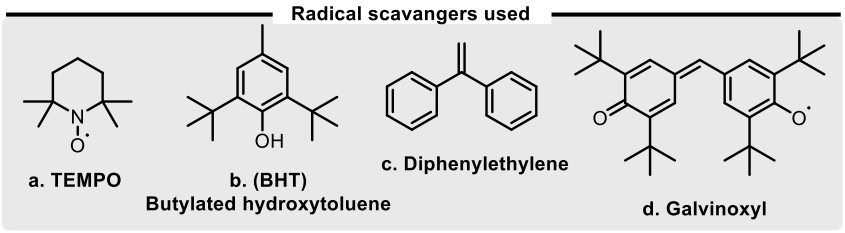
Scheme S15: General procedures for the radical quenching experiments.



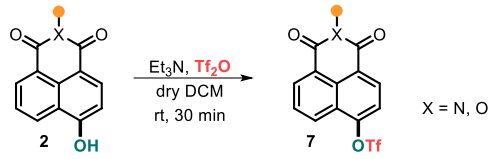
In an oven-dried pressure tube equipped with a magnetic stir bar, were placed *N*-substituted naphthalene monoimides **1d** (0.1 mmol, 1 equiv.) cobalt diacetate tetrahydrate (7 mg, 0.03 mmol, 0.3 equiv.), ammonium persulfate (34 mg, 0.15 mmol, 1.5 equiv.) and the radical scavenger (0-10 equiv.). Trifluoroacetic anhydride (0.2 mL) and trifluoroacetic acid (0.1 mL) with a volume ratio of 2:1 were added. The tube was fitted with a Teflon screw cap and the reaction mixture was stirred at 60 ^{o}C in a paraffin oil bath for 12–18 h. After that, the reaction mixture was cooled to room temperature, and diluted with ethyl acetate (75 mL). The trifluoroacetic acid was quenched with aqueous solution of saturated sodium bicarbonate (25 mL). The organic layer was washed with water and dried over anhyd. Na_2SO_4. The solution was filtered and concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:4) or (1:1) as eluent.

Table S7. Reaction efficiency with various radical scavengers.

Entry	Radical scavengers	Yield (%)
1	a (1 equiv.)	30%
2	a (10 equiv.)	10%
3	b (2 equiv.)	0%
4	c (2 equiv.)	0%
5	d (2 equiv.)	0%



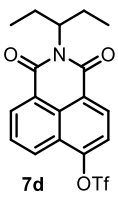
Scheme S16: General procedure for the synthesis of 4-trifluoromethane sulphonyl *N*-substituted-1,8-naphthalimides and 1,8-naphthalic anhydride (7):



In an oven-dried RB flask, 4-hydroxy-1,8-naphthalimide or 1,8-naphthalic anhydride (0.1 mmol, 1 equiv.) was dissolved in anhydrous DCM (1 mL). Then triethyl amine (60 µL, 0.2 mmol, 2 equiv.) and triflic anhydride (70 µL, 0.2 mmol, 2 equiv.) were added to the reaction mixture, sequentially, at 0 °C. The cooling bath was removed, and the reaction mixture was then stirred for 30 min at room temperature. After TLC analysis indicated the completion of the reaction, the reaction mixture was diluted with DCM (75 mL) and washed with water (25 mL). The organic layer was subsequently washed with saturated $NaHCO_3$ and brine, and dried over anhyd. Na_2SO_4. The solution was filtered and concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:19) or (1:9) as eluent to get the desired *peri*-trifluoromethane sulphonyl NMIs.

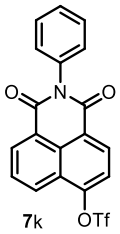
Analytical Data:

1,3-Dioxo-2-(pentan-3-yl)-2,3-dihydro-1*H*-benzo[*de*]isoquinolin-6-yl



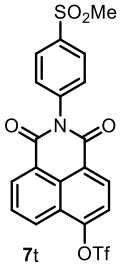
trifluoromethanesulfonate: Yield: 60% (25 mg); Physical appearance: white solid; TLC R_f 0.25 (Petroleum ether:Ethyl acetate, 19:1); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.71 (d, *J* = 7.0 Hz, 1H), 8.64 (d, *J* = 8.0 Hz, 1H), 8.42 (d, *J* = 8.5 Hz, 1H), 7.95 (t, *J* = 7.9 Hz, 1H), 7.75 (d, *J* = 8.1 Hz, 1H), 5.18 – 4.88 (m, 1H), 2.39 – 2.14 (m, 2H), 1.93 (m, 2H), 0.92 (t, *J* = 7.4 Hz, 6H); **^{13}C{^{1}H} NMR** (126 MHz, $CDCl_3$) δ 148.9, 129.7, 128.7, 126.7, 124.7, 119.9, 118.9, 117.4, 57.8, 24.9, 11.3; **19F NMR** (471 MHz, CDCl3) δ –72.83; **IR** (thin film, neat, cm^{-1}) 2203, 1688, 1576, 1316, 1089, 898; **ESI-HRMS:** Calculated for $C_{18}H_{17}F_3NO_5S^+$ $[M+H]^+$ 416.0774, found 416.0776.

1,3-dioxo-2-phenyl-2,3-dihydro-1*H*-benzo[*de*]isoquinolin-6-yl trifluoromethanesulfonate; Yield: 47% (20 mg); Physical appearance: white solid; TLC R_f 0.30 (9:1, Petroleum ether: EA);

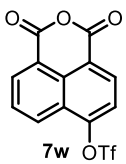


^{1}H NMR (500 MHz, $CDCl_3$) δ 8.78 (d, *J* = 7.3 Hz, 1H), 8.70 (d, *J* = 8.2 Hz, 1H), 8.50 (d, *J* = 8.5 Hz, 1H), 7.99 (t, *J* = 8.06 Hz, 1H), 7.80 (d, *J* = 8.1 Hz, 1H), 7.59 (t, *J* = 7.6 Hz, 2H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.34 (d, *J* = 7.7 Hz, 2H); **^{13}C{^{1}H} NMR** (126 MHz, $CDCl_3$) δ 163.6, 162.9, 149.4, 134.9, 134.9, 132.8, 131.7, 129.9, 129.5, 128.9, 128.9, 128.5, 127.3, 125.0, 123.3, 122.9, 122.5, 119.9, 119.1, 118.7 (q, J_{C-F} = 320.2 Hz), 117.4, 114.9; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –72.78; **IR** (thin film, neat, cm^{-1}) 2728, 1680, 1588, 1334, 1316, 1224, 1051, 648; **ESI-HRMS**: Calculated for $C_{19}H_{10}F_3NO_5SNa^+$ $[M+Na]^+$ 444.0124, found 444.0099.

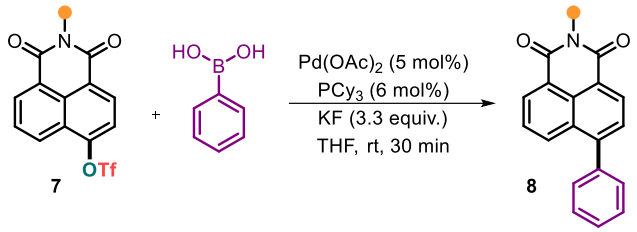
2-(4-(methylsulfonyl)phenyl)-1,3-dioxo-2,3-dihydro-1*H*-benzo[*de*]isoquinolin-6-yl trifluoromethanesulfonate; Yield: 74% (37 mg); Physical appearance: white solid; TLC R_f 0.30 (9:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.80 (d, *J* = 7.2 Hz, 1H), 8.73 (d, *J* = 8.2 Hz, 1H), 8.54 (d, *J* = 8.5 Hz, 1H), 8.17 (d, *J* = 8.5 Hz, 2H), 8.02 (t, *J* = 7.9 Hz, 1H), 7.83 (d, *J* = 8.1 Hz, 1H), 7.58 (d, *J* = 8.4 Hz, 2H), 3.16 (s, 3H); **^{13}C{^{1}H} NMR** (176 MHz, $CDCl_3$) δ 163.3, 162.6, 149.7, 140.9, 139.8, 133.2, 132.1, 130.1, 129.9, 128.9, 128.8, 127.9, 125.2, 122.8, 122.4, 119.3, 44.6; **^{19}F NMR** (471 MHz, $CDCl_3$) δ -72.72; **IR** (thin film, neat, cm^{-1}) 2728, 2229, 1680, 1325, 1314, 1220, 1129, 1108, 987; **ESI-HRMS**: Calculated for $C_{20}H_{12}F_3NO_7S_2Na^+$ $[M+Na]^+$ 521.9899, found 521.9892.



1,3-Dioxo-1*H*,3*H*-benzo[de]isochromen-6-yl trifluoromethanesulfonate; Yield: 78% (27 mg); **Physical appearance**: white solid; TLC R_f 0.35 (4:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.79 (d, *J* = 7.3 Hz, 1H), 8.72 (d, *J* = 8.1 Hz, 1H), 8.55 (d, *J* = 8.5 Hz, 1H), 8.04 (t, *J* = 7.9 Hz, 1H), 7.84 (d, *J* = 8.1 Hz, 1H); **^{13}C{^{1}H} NMR** (126 MHz, $CDCl_3$) δ 159.6, 159.0, 150.0, 134.7, 133.7, 131.6, 129.3, 128.5, 125.2, 119.9, 119.5, 119.4, 118.9, 117.4; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –72.65; **IR** (thin film, neat, cm^{-1}) 2120, 1683, 1598, 1370, 1056, 852; **ESI-HRMS**: Calculated for $C_{13}H_6F_3O_6S^+$ $[M+H]^+$ 346.9832, found 346.9827.

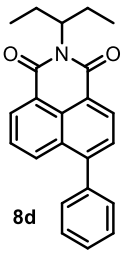


Scheme S17: General procedure for the synthesis of 4-aryl-1,8-naphthalimides (9).



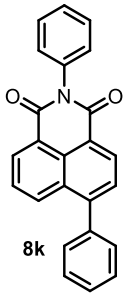
In an oven-dried RB flask, 4-trifluotomethane sulphonyl-1,8-naphthalimide (0.1 mmol, 1 equiv.), phenylboronic acid (15 mg, 0.12 mmol, 1.2 equiv.), $Pd(OAc)_2$ (1 mg, 0.005 mmol, 0.05 equiv.), PCy_3 (2 mg, 0.006 mmol, 0.06 equiv.), KF (19 mg, 0.33 mmol, 3.3 equiv.) were taken. Then anhydrous THF (0.3 mL) was added to the reaction mixture under inert atmosphere and the reaction mixture was stirred for 30 min at room temperature. After TLC analysis indicated the completion of the reaction, the solvent was evaporated under reduced pressure and the residue was diluted with DCM and passed through a Celite column. The filtrate was washed with brine (25 mL), dried over anhyd. Na_2SO_4 and filtered. This filtrate was concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:19) or (1:9) as eluent to get the desired 4-aryl NMIs.

Analytical Data:


8d

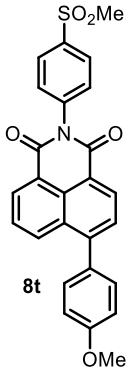
2-(Pentan-3-yl)-6-phenyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 81% (28 mg); Physical appearance: white solid; TLC R_f 0.40 (98:2, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.73 – 8.58 (m, 2H), 8.28 (d, *J* = 8.4 Hz, 1H), 7.73 (d, *J* = 7.6 Hz, 2H), 7.64 – 7.46 (m, 5H), 5.21 – 5.00 (m, 1H), 2.41 – 2.17 (m, 2H), 2.05 – 1.84 (m, 2H), 0.94 (t, *J* = 7.4 Hz, 6H); **^{13}C{^{1}H} NMR** (126 MHz, $CDCl_3$) δ 146.6, 138.9, 132.3, 129.9, 129.9, 128.9, 128.7, 128.4, 127.9, 126.9, 57.4, 25.1, 11.3; **IR** (thin film, neat, cm^{-1}) 2107, 1678, 1543, 1429, 1220, 1034, 751; **ESI-HRMS**: Calculated for $C_{23}H_{22}NO_2^+$ $[M+H]^+$ 344.1645, found 344.1628.

2,6-Diphenyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 93% (32 mg); Physical



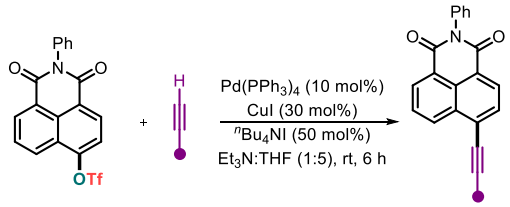
appearance: white solid; TLC R_f 0.25 (9:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.72 (t, *J* = 7.3 Hz, 2H), 8.36 (d, *J* = 8.5 Hz, 1H), 7.76 (t, *J* = 7.5 Hz, 2H), 7.66-7.49 (m, 8H), 7.38 (d, J = 7.3 Hz, 2H); **^{13}C{^{1}H} NMR** (126 MHz, $CDCl_3$) δ 164.5, 164.3, 147.3, 138.8, 135.5, 133.1, 131.6, 131.2, 130.3, 129.9, 129.4, 129.1, 128.7, 128.7, 128.6, 127.9, 126.9, 123.0, 121.9; **IR** (thin film, neat, cm^{-1}) 1694, 1720, 1634, 1330, 1267, 1005, 848; **ESI-HRMS**: Calculated for $C_{24}H_{16}NO_2^+$ $[M+H]^+$ 350.1176, found 350.1157.

6-(4-methoxyphenyl)-2-(4-(methylsulfonyl)phenyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-



dione; Yield: 98% (45 mg); Physical appearance: white solid; TLC R_f 0.10 (4:1, DCM: MeOH); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.71 (d, *J* = 7.5 Hz, 2H), 8.44 (d, *J* = 8.8 Hz, 1H), 8.17 (d, *J* = 8.4 Hz, 2H), 7.77 (d, *J* = 7.5 Hz, 2H), 7.61 (d, *J* = 8.5 Hz, 2H), 7.51 (d, *J* = 8.6 Hz, 2H), 7.14 (d, *J* = 8.6 Hz, 2H), 3.96 (s, 3H), 3.17 (s, 3H); **^{13}C{^{1}H} NMR** Owing to the solubility issue, ^{13}C NMR for this compound could not be recorded; **IR** (thin film, neat, cm^{-1}) 2930, 2863, 1680, 1627, 1582, 1499, 1421, 1370, 1265, 1230, 1024, 845; **ESI-HRMS**: Calculated for $C_{26}H_{20}NO_5S^+$ $[M+H]^+$ 458.1057, found 458.1059.

Scheme S18: General procedure for the synthesis of 4-alkynyl-1,8-naphthalimides (9 & 10):

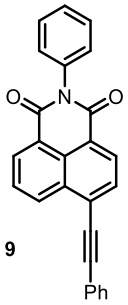


In an oven-dried RB flask, 4-trifluotomethane sulphonyl-1,8-naphthalimide (0.1 mmol, 1 equiv.), phenyl acetylene (12 mg, 0.12 mmol, 1.2 equiv.), $Pd(PPh_3)_4$ (11 mg, 0.01 mmol, 0.1 equiv.), CuI (6 mg, 0.03 mmol, 0.3 equiv.), tetrabutylammonium iodide (18 mg, 0.05 mmol, 0.5 equiv.) were taken. Anhydrous THF (0.3 mL) and triethyl amine (60 μL) were added to the reaction mixture under an inert atmosphere and the reaction mixture was stirred for 6 h at room temperature. After

TLC analysis indicated the completion of the reaction, the solvent was evaporated under reduced pressure and the residue was diluted with DCM and passed through a Celite column. The filtrate was washed with brine (25 mL) and dried over anhyd. Na_2SO_4. This was filtered and concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:4) as eluent to get the desired 4-alkynyl NMIs.

Analytical Data:

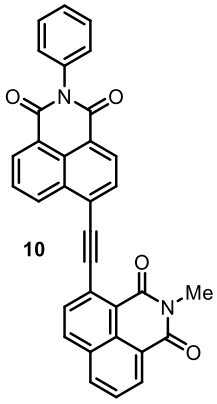
2-Phenyl-6-(phenylethynyl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 91% (34 mg);



9

Physical appearance: yellow solid; TLC R_f 0.20 (1:4, Petroleum ether: EA); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.84 (d, *J* = 7.7 Hz, 1H), 8.72 (d, *J* = 7.2 Hz, 1H), 8.63 (d, *J* = 7.6 Hz, 1H), 8.02 (d, *J* = 7.6 Hz, 1H), 7.95 – 7.86 (t, *J* = 7.8 Hz, 1H), 7.77 – 7.68 (m, 2H), 7.59 (t, *J* = 7.5 Hz, 2H), 7.53 (d, *J* = 7.3 Hz, 1H), 7.51 – 7.43 (m, 3H), 7.39 – 7.32 (m, 2H); **^{13}C{^{1}H} NMR** (101 MHz, CDCl3) δ 164.2, 163.9, 135.3, 132.8, 132.0, 131.9, 131.8, 130.8, 130.8, 129.5, 129.4, 128.8, 128.7, 128.6, 128.5, 128.1, 127.6, 123.1, 122.2, 99.4, 86.3; **IR** (thin film, neat, cm^{-1}) 2918, 2950, 1710, 1669, 1589, 1364, 1462, 1238, 1191, 756; **ESI-HRMS**: Calculated for $C_{26}H_{16}NO_2^+$ $[M+H]^+$ 374.1176, found 374.1170.

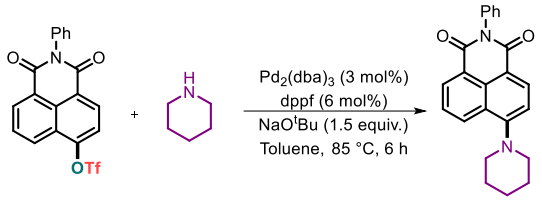
4-((1,3-Dioxo-2-phenyl-2,3-dihydro-1*H*-benzo[*de*]isoquinolin-6-yl)ethynyl)-2-methyl-1*H*-



10

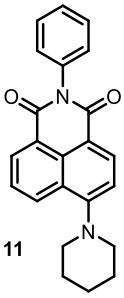
benzo[de]isoquinoline-1,3(2*H*)-dione; Yield: 93% (45 mg); Physical appearance: yellow solid; TLC R_f 0.30 (1:1, Petroleum ether: EA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.42 (d, *J* = 8.4 Hz, 1H), 8.80 – 8.72 (m, 2H), 8.68 (d, *J* = 7.6 Hz, 1H), 8.32-8.24 (m, 2H), 8.21 (d, *J* = 7.6 Hz, 1H), 8.09-8.00 (m, 2H), 7.86 (t, *J* = 7.8 Hz, 1H), 7.60 (t, *J* = 7.6 Hz, 2H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.38 (d, *J* = 7.3 Hz, 2H), 3.70 (s, 3H); **^{13}C{^{1}H} NMR** (176 MHz, $CDCl_3$) δ 164.2, 163.9, 163.8, 163.1, 135.3, 133.9, 133.7, 133.0, 132.6, 132.5, 132.2, 132.1, 131.9, 131.4, 130.7, 129.4, 128.8, 128.6, 128.5, 128.5, 128.1, 127.8, 127.7, 125.7, 123.2, 123.1, 122.9, 122.6, 98.9, 96.0, 27.3; **IR** (thin film, neat, cm^{-1}) 2922, 2851, 1705, 1663, 1587, 1369, 1236; **ESI-HRMS**: Calculated for $C_{33}H_{19}N_2O_4^+$ $[M+H]^+$ 507.1339, found 507.1337.

Scheme S19: General procedure for the synthesis of 4-amino-1,8-naphthalimides (11):



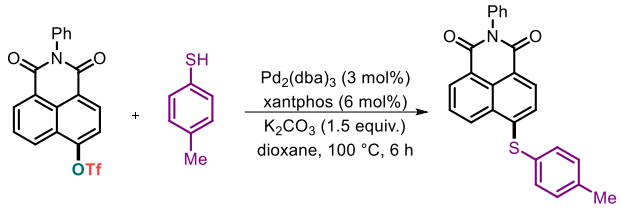
In an oven-dried pressure tube, 4-trifluotomethane sulphonyl-1,8-naphthalimide (0.1 mmol, 1 equiv.), $Pd_2(dba)_3$ (3 mg, 0.003 mmol, 0.03 equiv.), 1,1'-bis(diphenylphosphino)ferrocene ligand (3 mg, 0.006 mmol, 0.06 equiv.), sodium *tert*-butoxide (14 mg, 0.15 mmol, 1.5 equiv.) were taken. Anhydrous toluene (0.3 mL) was added to the reaction mixture under an inert atmosphere and the reaction mixture was stirred for 5 min at room temperature. Then piperidine (15 μL, 13 mg, 0.15 mmol, 1.5 equiv.) was added to the reaction mixture and the reaction mixture was stirred at 85 ^oC in a paraffin oil bath for 6 h. After TLC analysis indicated the completion of the reaction, the THF was evaporated from the reaction mixture and diluted with DCM and passed through a Celite column. The organic layer (75 mL) was washed with brine (25 mL) and dried over anhyd. Na_2SO_4. This solution was filtered and concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:4) as eluent to get the desired 4-amino NMIs.

2-Phenyl-6-(piperidin-1-yl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 82% (29 mg); Physical appearance: yellow solid; TLC R_f 0.30 (1:4, Petroleum ether: EA); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.64 (d, *J* = 6.7 Hz, 1H), 8.57 (d, *J* = 8.1 Hz, 1H), 8.51 (d, *J* = 8.4 Hz, 1H), 7.75 (t, *J* = 8.0 Hz, 1H), 7.57 (t, *J* = 7.5 Hz, 2H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.36 – 7.30 (m, 2H), 7.28–7.22 (m, 1H), 3.39 – 3.23 (m, 4H), 2.03 – 1.90 (m, 4H), 1.78 (m, 2H); **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 164.8, 164.3, 135.7, 133.0, 131.5, 131.0, 130.4, 129.3, 128.7, 128.5, 126.4, 125.5, 123.3, 116.1, 114.9, 54.7, 26.2, 24.3; **IR** (thin film, neat, cm^{-1}) 2928, 1704, 1650, 1591, 1575, 1372, 1357, 1232, 1188, 1139, 997, 780; **ESI-HRMS**: Calculated for $C_{23}H_{22}N_2O_2^+$ $[M+H]^+$ 357.1598, found 357.1594.



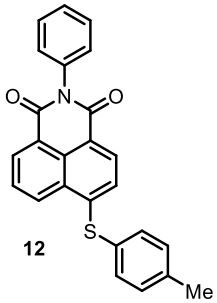
11

Scheme S20: General procedure for the synthesis of 4-thio-1,8-naphthalimides (12):



In an oven-dried sealed tube, 4-trifluoromethane sulphonyl-1,8-naphthalimide (0.1 mmol, 1 equiv.), $Pd_2(dba)_3$ (3 mg, 0.003 mmol, 0.03 equiv.), xantphos ligand (3 mg, 0.006 mmol, 0.06 equiv.) were taken. In another 5 mL RB flask, *para*-methyl thiophenol (19 mg, 0.15 mmol, 1.5 equiv.) and potassium carbonate (21 mg, 0.15 mmol, 1.5 equiv.) were taken and dissolved in anhydrous dioxane (0.3 mL). This solution was added to the reaction mixture in the sealed tube, passing it through a syringe filter. The resulting reaction mixture was sealed with a Teflon screw cap and stirred for 6 h at 100 ^oC in a paraffin oil bath. After TLC analysis indicated the completion of the reaction, the reaction mixture was diluted with DCM and passed through a Celite column. The filtrate was concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:4) as eluent to get the desired 4-(*p*-tolylthio) NMI.

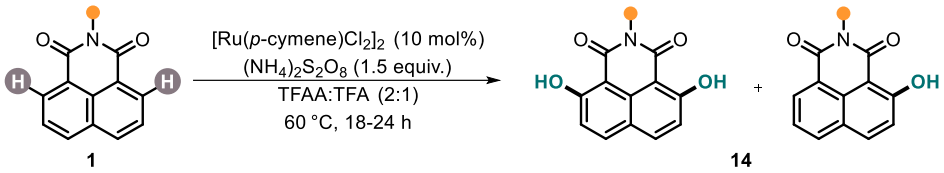
2-Phenyl-6-(*p*-tolylthio)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 60% (24 mg);



12

Physical appearance: yellow solid; TLC R_f 0.40 (4:1, Petroleum ether: EA); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.72 (t, *J* = 7.9 Hz, 2H), 8.39 (d, *J* = 7.9 Hz, 1H), 7.85 (t, *J* = 7.9 Hz, 1H), 7.63-7.54 (m, 2H), 7.54-7.45 (m, 3H), 7.33 (d, *J* = 7.8 Hz, 4H), 7.22 (d, *J* = 7.9 Hz, 1H), 2.46 (s, 3H); **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 164.2, 164.1, 147.0, 140.2, 135.4, 134.9, 132.0, 131.2, 131.0, 131.0, 130.4, 129.5, 129.4, 129.1, 128.8, 128.6, 128.5, 126.9, 126.4, 124.7, 123.3, 119.7, 21.3; **IR** (thin film, neat, cm^{-1}) 2921, 1707, 1666, 1585, 1491, 1364, 1350, 1236, 1191, 1133, 972, 777; **APCI-HRMS**: Calculated for $C_{25}H_{18}NO_2S^+$ $[M+H]^+$ 396.1053, found 396.1051.

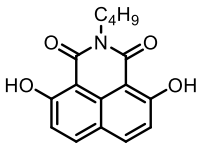
Scheme S21: General procedure for the synthesis of *ortho*-hydroxy-*N*-alkyl NMIs (14).



In an oven-dried pressure tube equipped with a magnetic stir bar, was placed *N*-alkyl naphthalene monoimide (0.1 mmol, 1 equiv.) [Ru(*p*-cymene)Cl_2]$_2$ (6 mg, 0.01 mmol, 0.1 equiv.) and ammonium persulfate (34 mg, 0.15 mmol, 1.5 equiv.). Then trifluoroacetic anhydride (0.2 mL) and trifluoroacetic acid (0.1 mL) with volume ratio 2:1 were added. The tube was fitted with a Teflon screw cap and the reaction mixture was stirred at 60 ^{o}C in a paraffin oil bath for 12-18 h. After TLC analysis indicated the completion of the reaction, the reaction mixture was cooled to room temperature, diluted with ethyl acetate (75 mL). The trifluoroacetic acid was quenched with an aqueous solution of saturated sodium bicarbonate (25 mL) and extracted. The organic layer was washed with water and dried over anhyd. Na_2SO_4. The solution was filtered and concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:19) or (2:98) as eluent to get the desired *ortho*-hydroxylated NMIs.

Analytical Data:

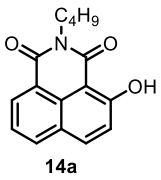
2-Butyl-4,9-dihydroxy-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 53% (15 mg);



14aa

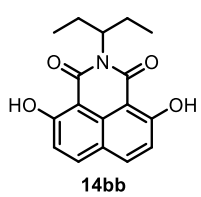
Physical appearance: white solid; TLC R_f 0.50 (EA:Hexane, 1:19); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.96 (s, 2H), 7.96 (d, *J* = 8.9 Hz, 2H), 7.12 (d, *J* = 8.9 Hz, 2H), 4.29 – 4.12 (m, 2H), 1.82 – 1.69 (m, 2H), 1.54 – 1.42 (m, 2H), 1.02 (t, *J* = 7.4 Hz, 3H); **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 168.2, 166.0, 137.4, 129.7, 119.3, 116.4, 102.0, 39.4, 29.9, 20.3, 13.8; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{16}H_{14}NO_4^-$ 284.0917, found 284.0913; **IR** (thin film, neat, cm^{-1}) 2956, 1623, 1657, 1470, 1400, 1370, 1296, 1261, 1222, 1194, 1160, 1083, 941, 822, 529.

2-Butyl-4-hydroxy-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 20% (5 mg); Physical appearance: white solid; TLC R_f 0.45 (1:19 = EA:Hexane); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.16 (s, 1H), 8.62 (d, *J* = 7.6 Hz, 1H), 8.12 (t, *J* = 9.0 Hz, 2H), 7.64 (t, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 9.1 Hz, 1H), 4.31 – 4.14 (m, 2H), 1.82 – 1.68 (m, 2H), 1.56 – 1.42 (m, 2H), 1.01 (t, *J* = 7.4 Hz, 3H); **^{13}C{^{1}H} NMR** (126 MHz, $CDCl_3$) δ 169.1, 165.5, 163.8, 136.9, 134.3, 131.7, 128.9, 126.0, 124.5, 120.9, 120.5, 102.0, 39.8, 30.1, 20.4, 13.8; **IR** (thin film, neat, cm^{-1}) 2959, 1681, 1637, 1592, 1440, 1475, 1406, 1356, 1196, 1173, 1081, 828, 754; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{16}H_{14}NO_3^-$ 268.0968, found 268.0955.



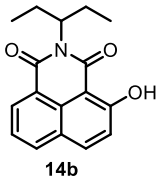
14a

4,9-Dihydroxy-2-(pentan-3-yl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 60% (18 mg); Physical appearance: white solid; TLC R_f 0.55 (EA:Hexane, 1:19); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 13.17 (s, 2H), 8.00 (d, *J* = 8.9 Hz, 2H), 7.15 (d, *J* = 8.9 Hz, 2H), 5.18– 5.06 (m, 1H), 2.39 – 2.20 (m, 2H), 2.04 – 1.87 (m, 2H), 0.92 (t, *J* = 7.5 Hz, 6H); **^{13}C{^{1}H} NMR** (126 MHz, $CDCl_3$) δ 166.2, 137.2, 130.1, 119.2, 116.5, 57.2, 24.8, 11.3; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{17}H_{16}NO_4^-$ 298.1074, found 298.1094; **IR** (thin film, neat, cm^{-1}) 2965, 2927, 2877, 1650, 1627, 1477, 1442, 1370, 1291, 1198, 1169, 1082, 830, 789.



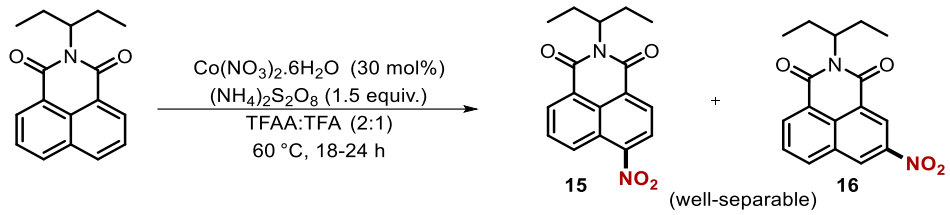
14bb

4-Hydroxy-2-(pentan-3-yl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 30% (8 mg); Physical appearance: white solid; TLC R_f 0.50 (EA:Hexane, 1:19); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 13.32 (s, 1H), 8.66 – 8.53 (m, 1H), 8.11 (t, *J* = 8.1 Hz, 2H), 7.63 (t, *J* = 7.7 Hz, 1H), 7.33 (d, *J* = 9.1 Hz, 1H), 5.18 – 5.01 (m, 1H), 2.36 – 2.19 (m, 2H), 2.02 – 1.86 (m, 2H), 0.92 (t, *J* = 7.5 Hz, 6H); **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 165.6, 136.7, 135.4, 134.0, 129.1, 126.9, 125.9, 124.5, 120.6, 24.9, 11.3; **IR** (thin film, neat, cm^{-1}) 2968, 2934, 2876, 1683, 1590, 1639, 1476, 1355, 1231, 1193, 1170, 1080, 824, 751; **ESI-HRMS:** $[M-H]^-$ Calculated for $C_{17}H_{16}NO_3^-$ 282.1125, found 282.1100.



14b

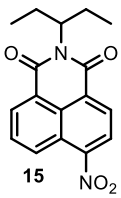
Scheme S22: General procedure for the synthesis of *bay*- and *peri*-nitrated *N*-alkyl 1,8-naphthalimide (15 & 16).



In an oven-dried pressure tube equipped with a magnetic stir bar were placed *N*-alkyl naphthalene monoimide (0.1 mmol, 1 equiv.) $Co(NO_3)_2.6H_2O$ (9 mg, 0.03 mmol, 0.3 equiv.) and ammonium persulfate (34 mg, 0.15 mmol, 1.5 equiv.). Trifluoroacetic anhydride (0.2 mL) and trifluoroacetic acid (0.1 mL) with a volume ratio 2:1 were added. The tube was fitted with a Teflon screw cap and the reaction mixture was stirred at 60 ^oC in a paraffin oil bath for 12-18 h. After TLC analysis indicated the completion of the reaction, the reaction mixture was cooled to room temperature, diluted with ethyl acetate (75 mL). The trifluoroacetic acid was quenched with aqueous solution of saturated sodium bicarbonate (25 mL) and extracted. The organic layer was washed with brine and dried over and Na_2SO_4. The solution was filtered and concentrated under reduced pressure and the residue was purified by silica gel flash column chromatography eluting with EA:Petroleum ether (1:10) as eluent to get the desired *bay*- and *peri*-nitrated NMIs.

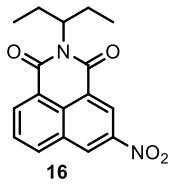
Analytical Data:

6-Nitro-2-(pentan-3-yl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 40% (12 mg);



Physical appearance: pale yellow solid; TLC R_f 0.40 (Petroleum ether:Ethyl acetate, 9:1); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.87 (d, *J* = 8.7 Hz, 1H), 8.74 (d, *J* = 7.3 Hz, 1H), 8.69 (d, *J* = 8.0 Hz, 1H), 8.44 (d, *J* = 8.0 Hz, 1H), 8.01 (t, *J* = 7.4 Hz, 1H), 5.10 – 4.99 (m, 1H), 2.32 – 2.17 (m, 2H), 2.03 – 1.87 (m, 2H), 0.93 (t, *J* = 7.5 Hz, 6H); **^{13}C{^{1}H} NMR** (126 MHz, $CDCl_3$) δ 149.3, 129.9, 129.3, 129.0, 123.9, 123.6, 58.1, 24.9, 11.3; **IR** (thin film, neat, cm^{-1}) 1970, 1842, 1688, 1276, 1208, 1022, 798; **ESI-HRMS** Calculated for $C_{17}H_{17}N_2O_4^+$ $[M+H]^+$ 313.1183, found 313.1188.

5-Nitro-2-(pentan-3-yl)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione; Yield: 57% (18 mg);



16

Physical appearance: pale yellow solid; TLC R_f 0.40 (Petroleum ether:Ethyl acetate, 9:1); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.32 (d, *J* = 1.6 Hz, 1H), 9.15 (d, *J* = 2.3 Hz, 1H), 8.78 (d, *J* = 7.3 Hz, 1H), 8.44 (d, *J* = 7.7 Hz, 1H), 7.96 (t, *J* = 7.7 Hz, 1H), 5.12 – 5.02 (m, 1H), 2.35 – 2.16 (m, 2H), 2.01 – 1.86 (m, 2H), 0.93 (t, *J* = 7.5 Hz, 6H); **^{13}C{^{1}H} NMR** (126 MHz, $CDCl_3$) δ 150.3, 146.5, 135.2, 130.9, 130.4, 129.4, 129.1, 128.6, 58.1, 24.9, 11.3; **IR** (thin film, neat, cm^{-1}) 1834, 1742, 1688, 1476, 1286, 1124, 951; **ESI-HRMS** Calculated for $C_{17}H_{17}N_2O_4^+$ $[M+H]^+$ 313.1183, found 313.1168.

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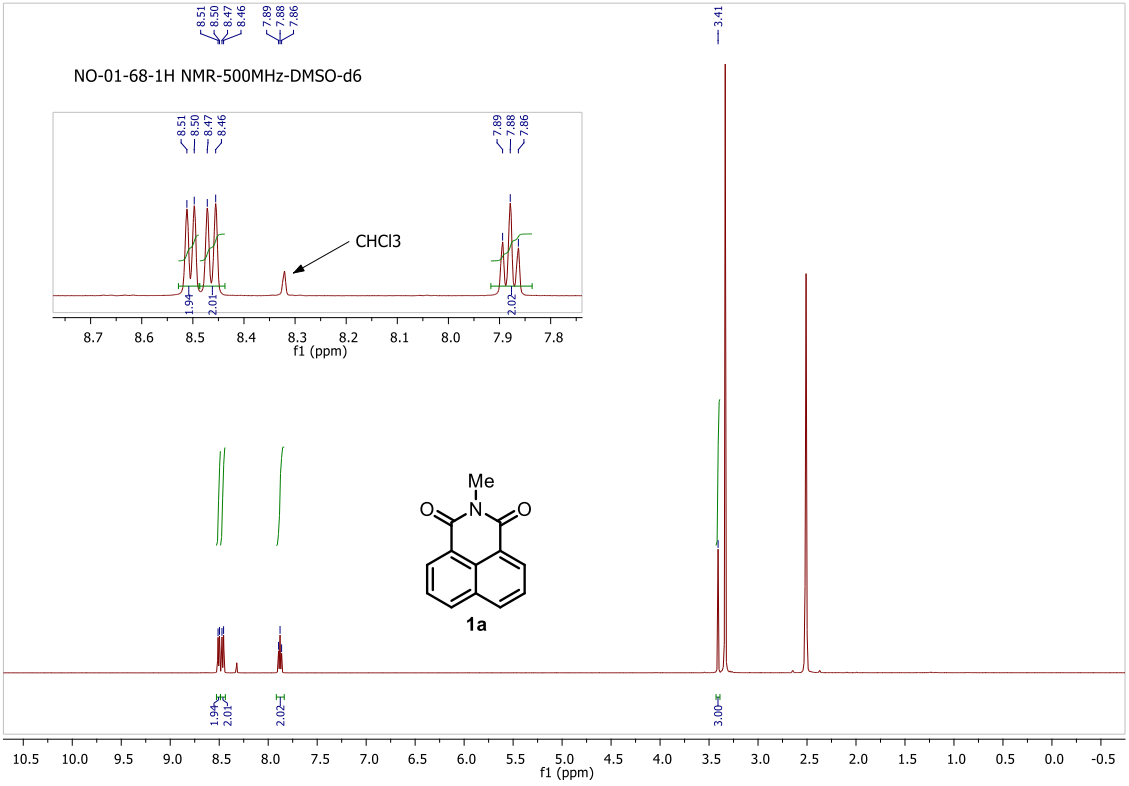
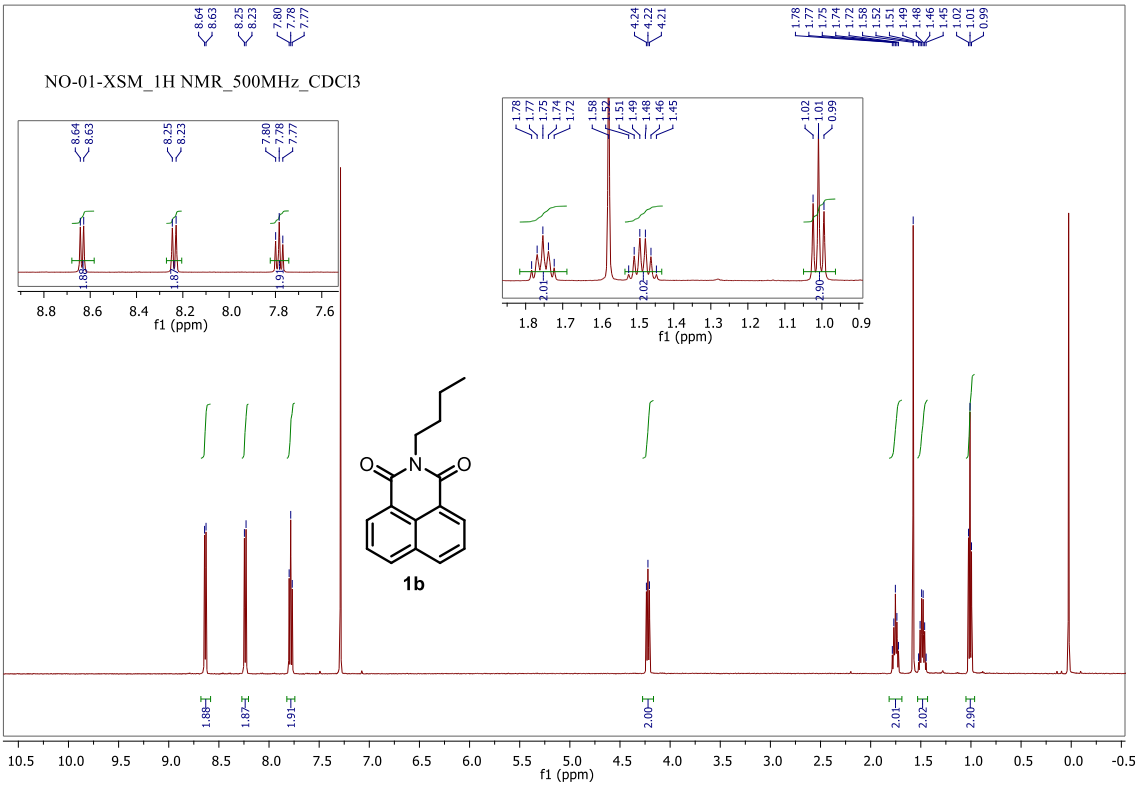
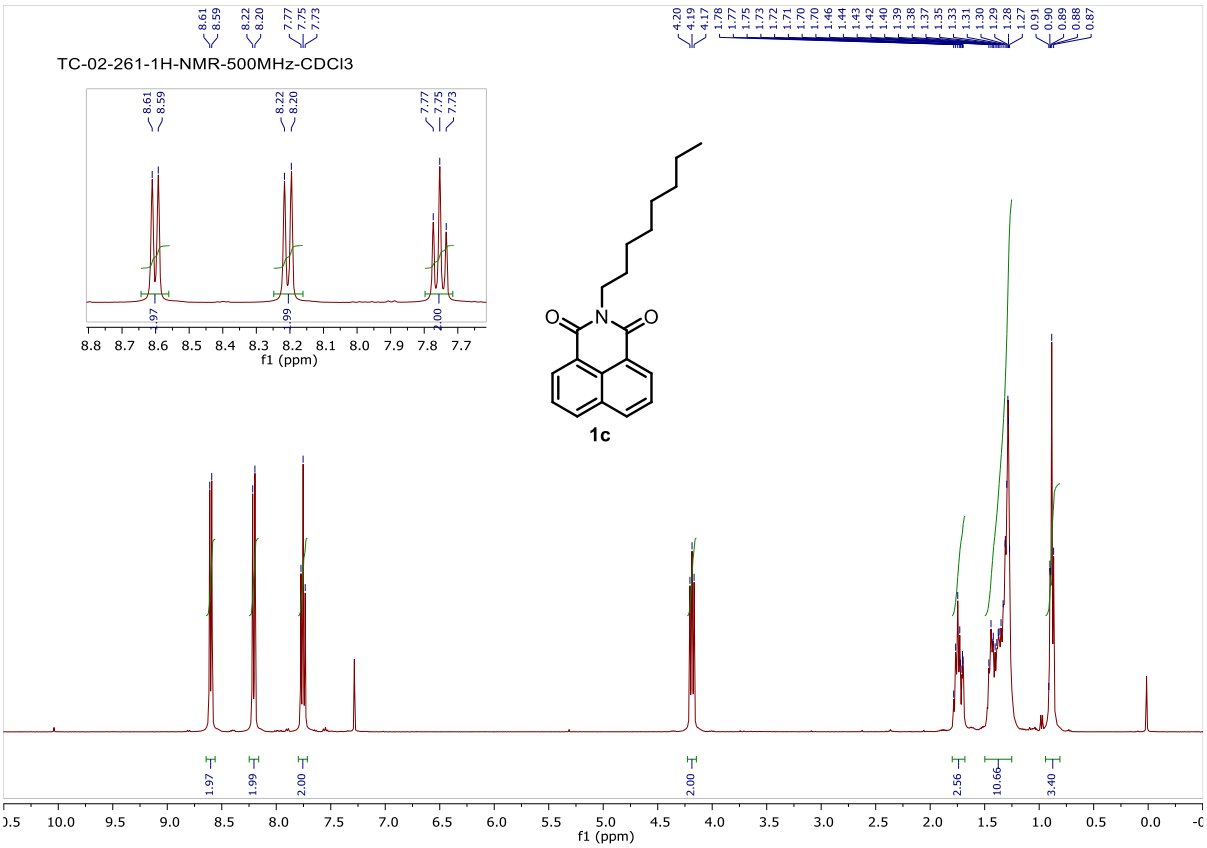
3. Copies of ^{1}H, ^{13}C and ^{19}F NMR and HRMS spectra:



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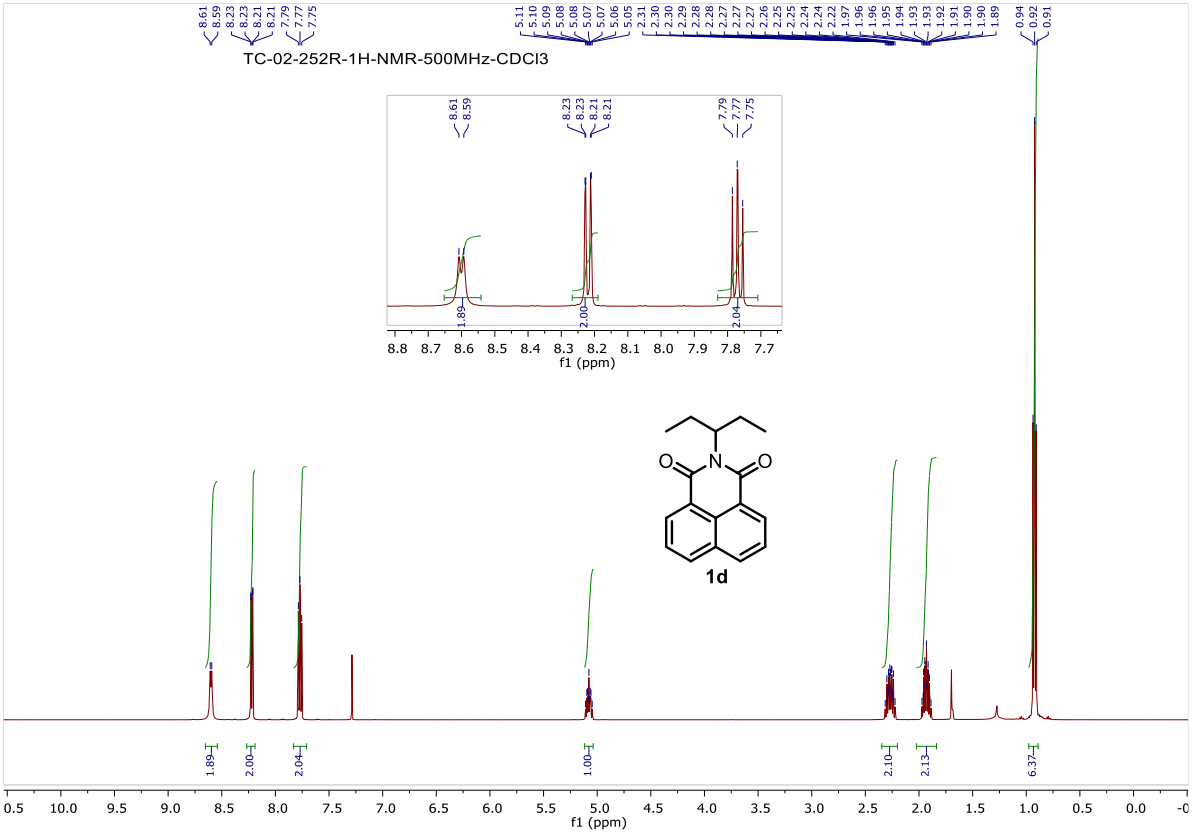
f1 (ppm)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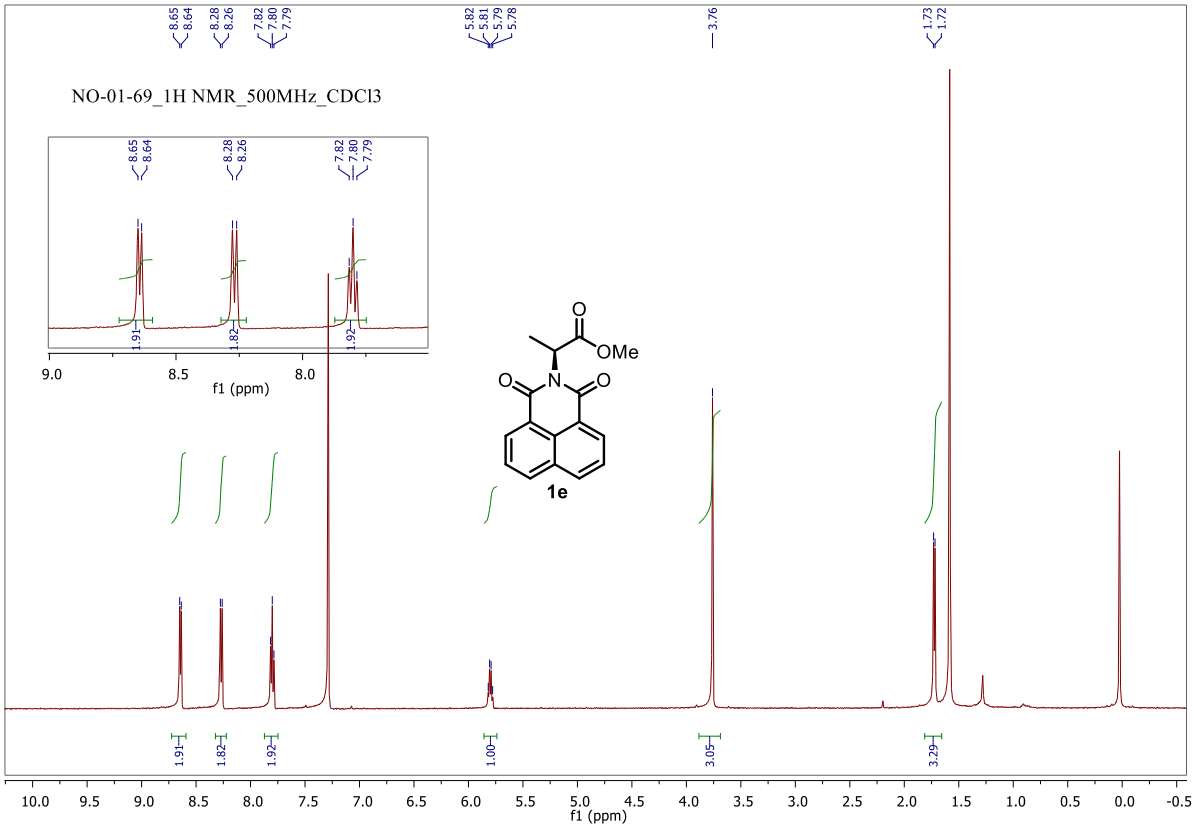
1d
f1 (ppm)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8.65
8.64
8.28
8.26
7.82
7.80
7.79
5.82
5.81
5.79
5.78
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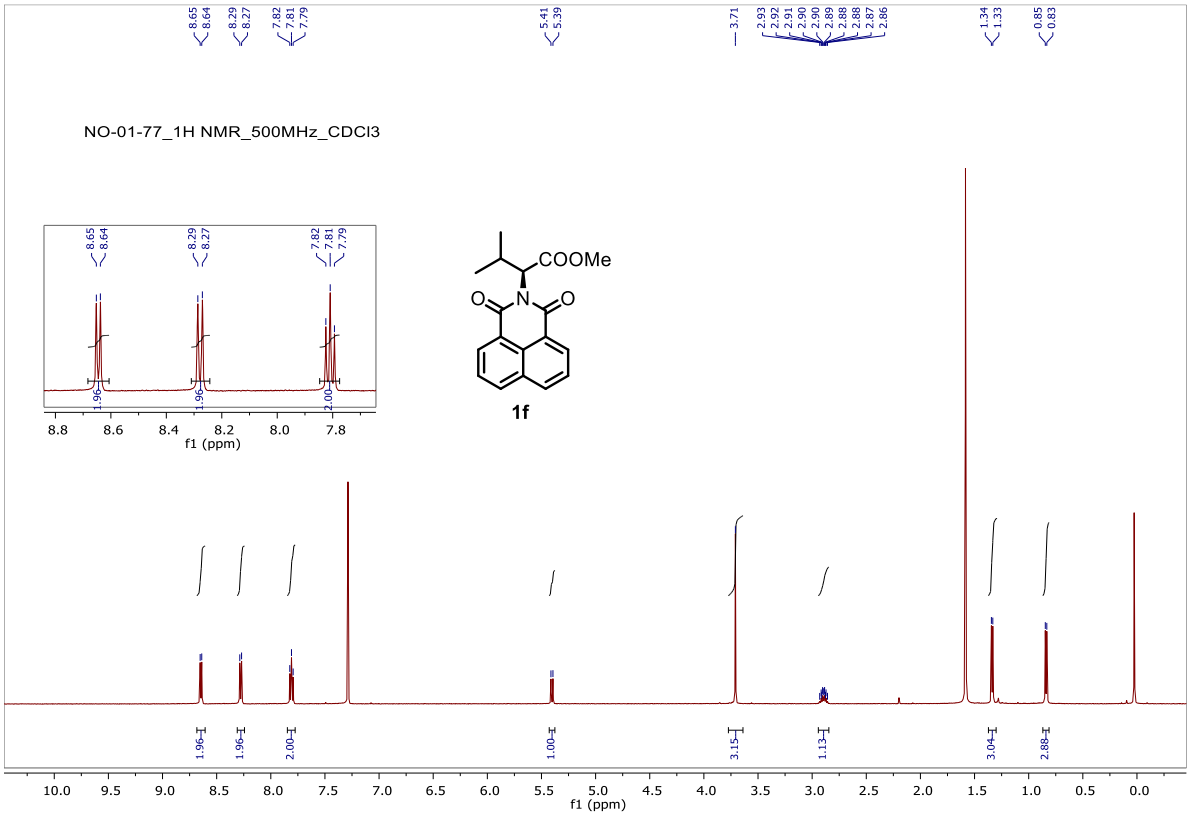
1.00
3.05
3.29
f1 (ppm)
OMe
1e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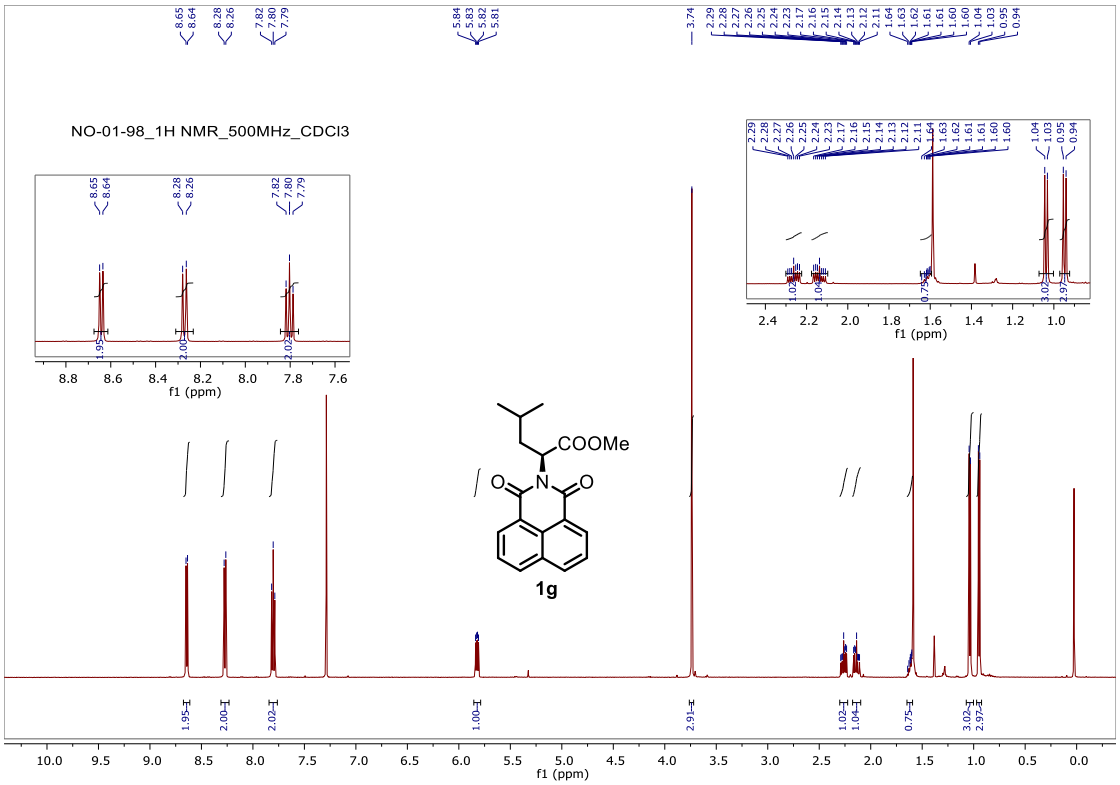
8.65
8.64
8.29
8.27
7.82
7.81
7.79
5.41
5.39
3.71
2.93
2.92
2.91
2.90
2.89
2.88
2.87
2.86
1.34
1.33
0.85
0.83
1.96
1.96
2.00
1.00
3.15
1.13
3.04
2.88
f1 (ppm)
COOMe
1f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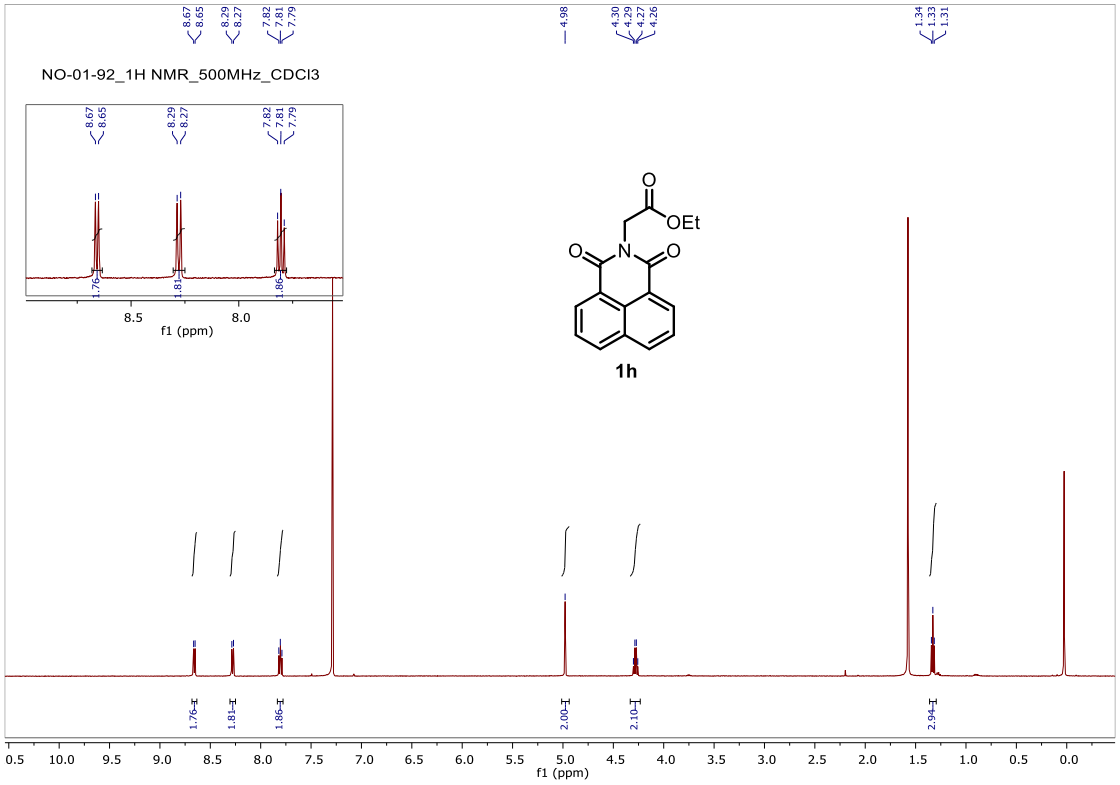
COOMe
1g
f1 (ppm)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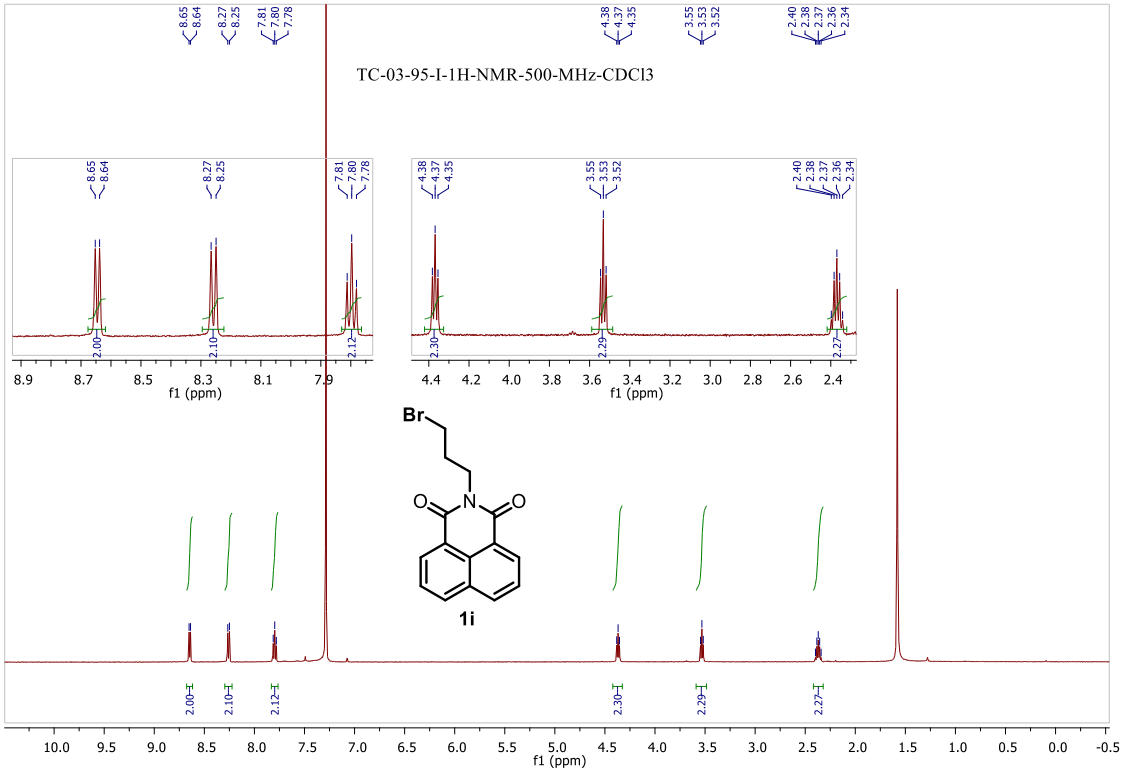
f1 (ppm)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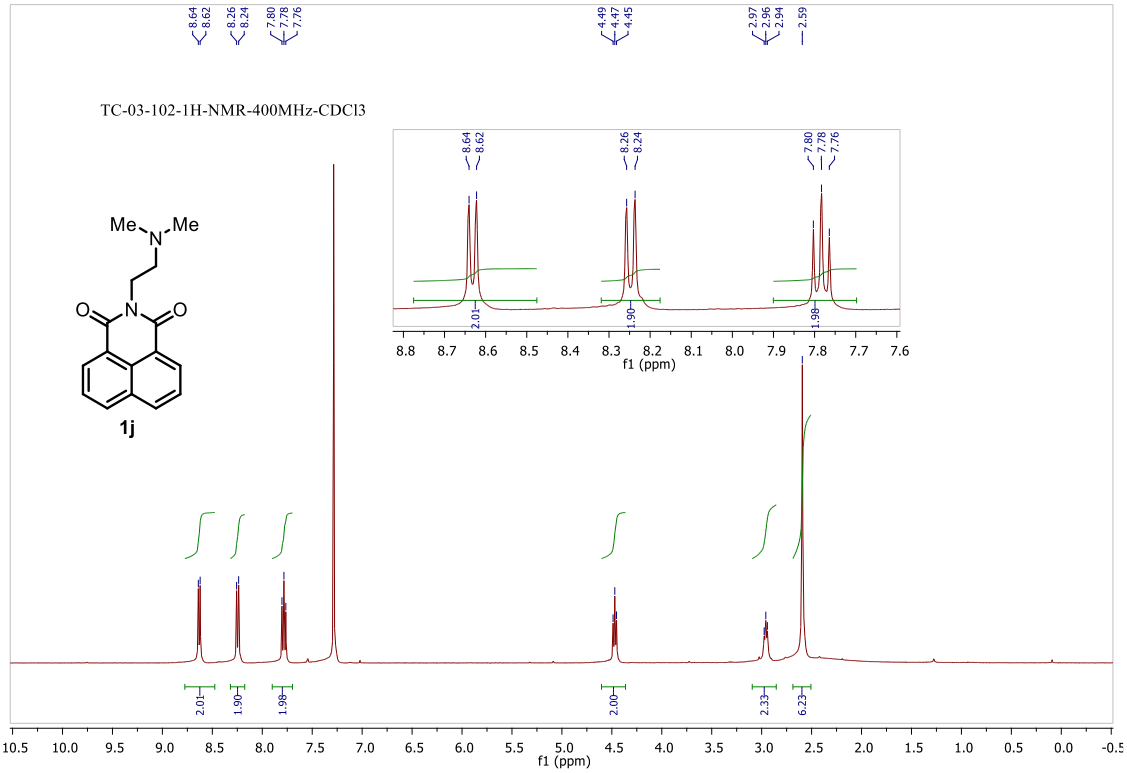
1i
f1 (ppm)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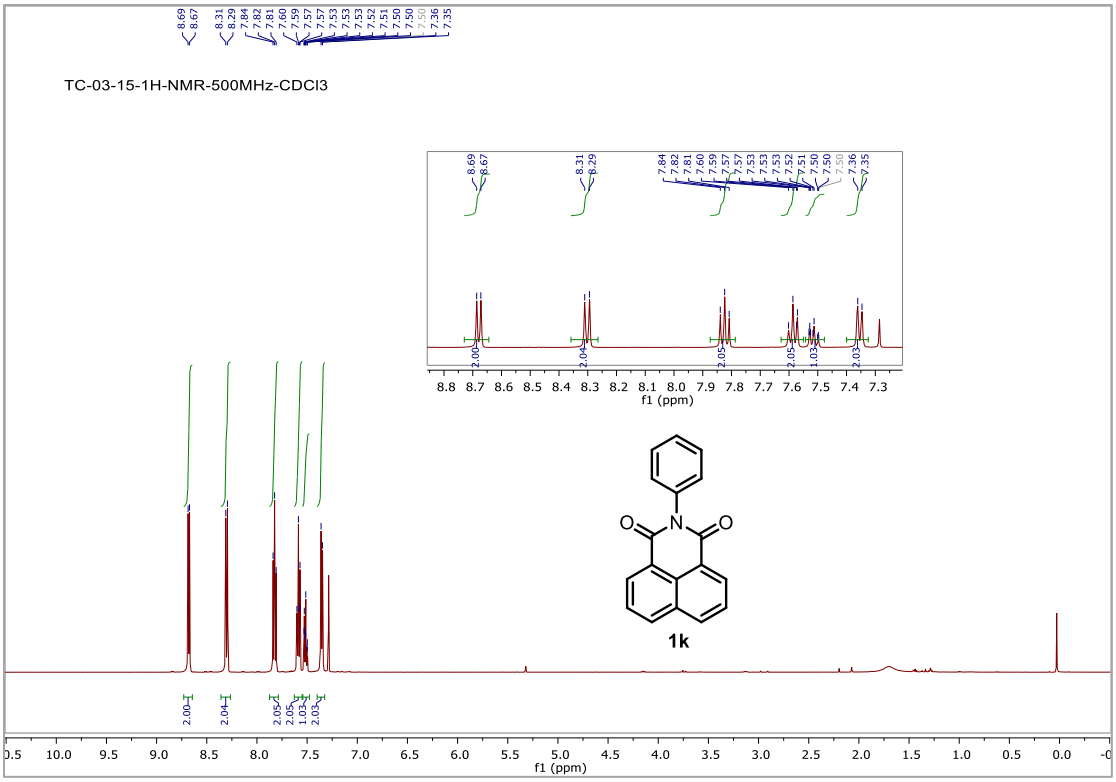
N
Me
1j
f1 (ppm)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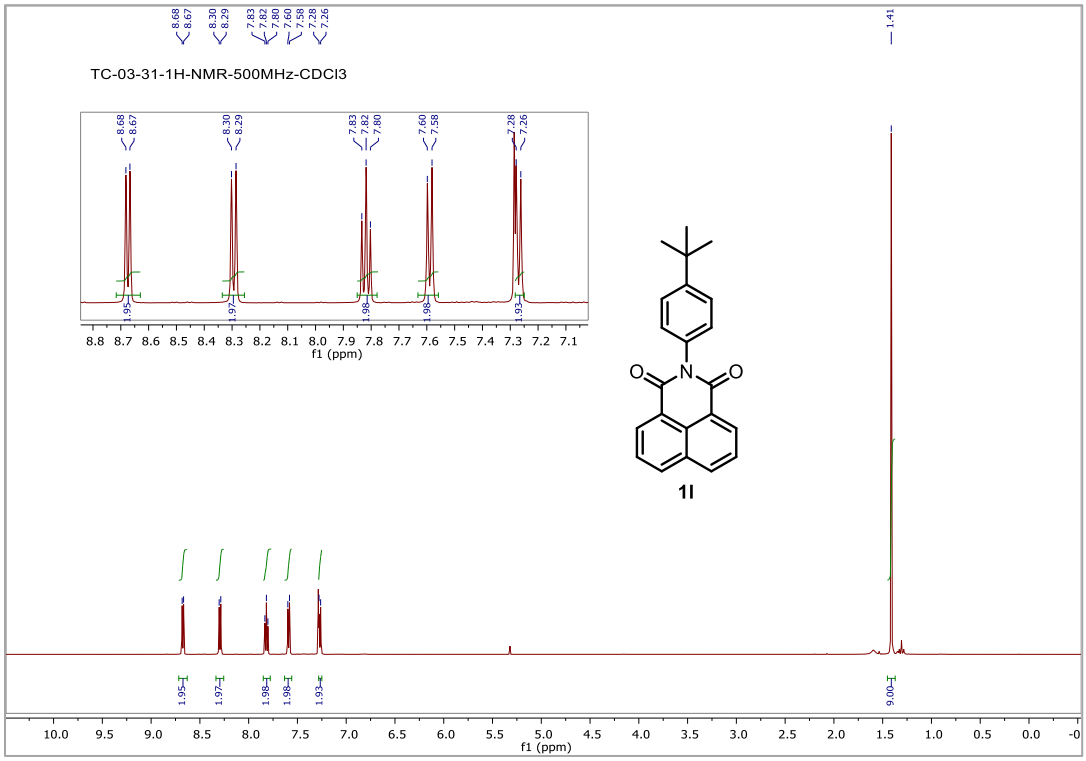
1k
f1 (ppm)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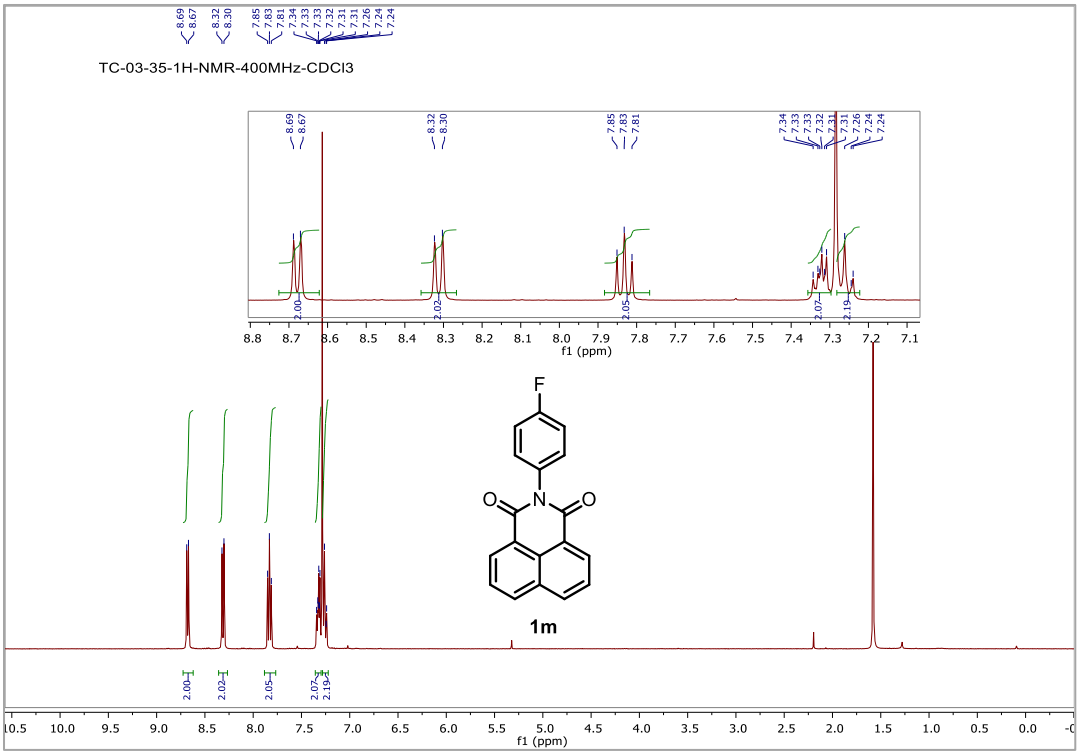
1l
f1 (ppm)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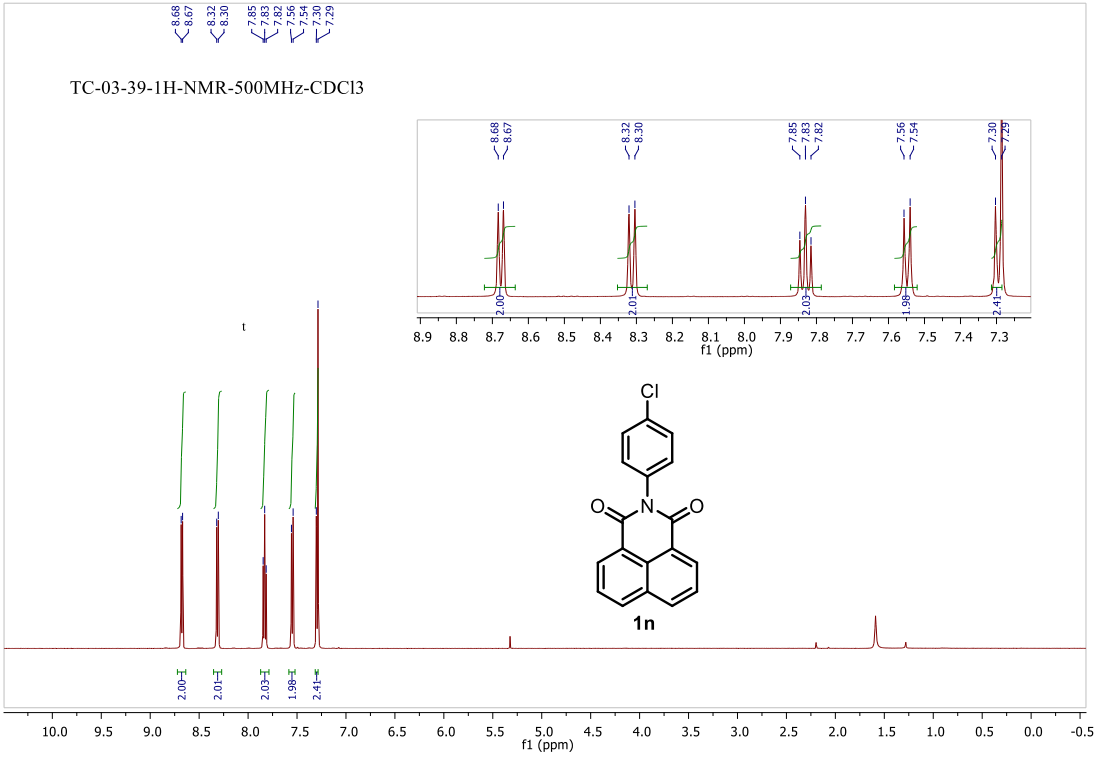
F
O
N
O
1m
f1 (ppm)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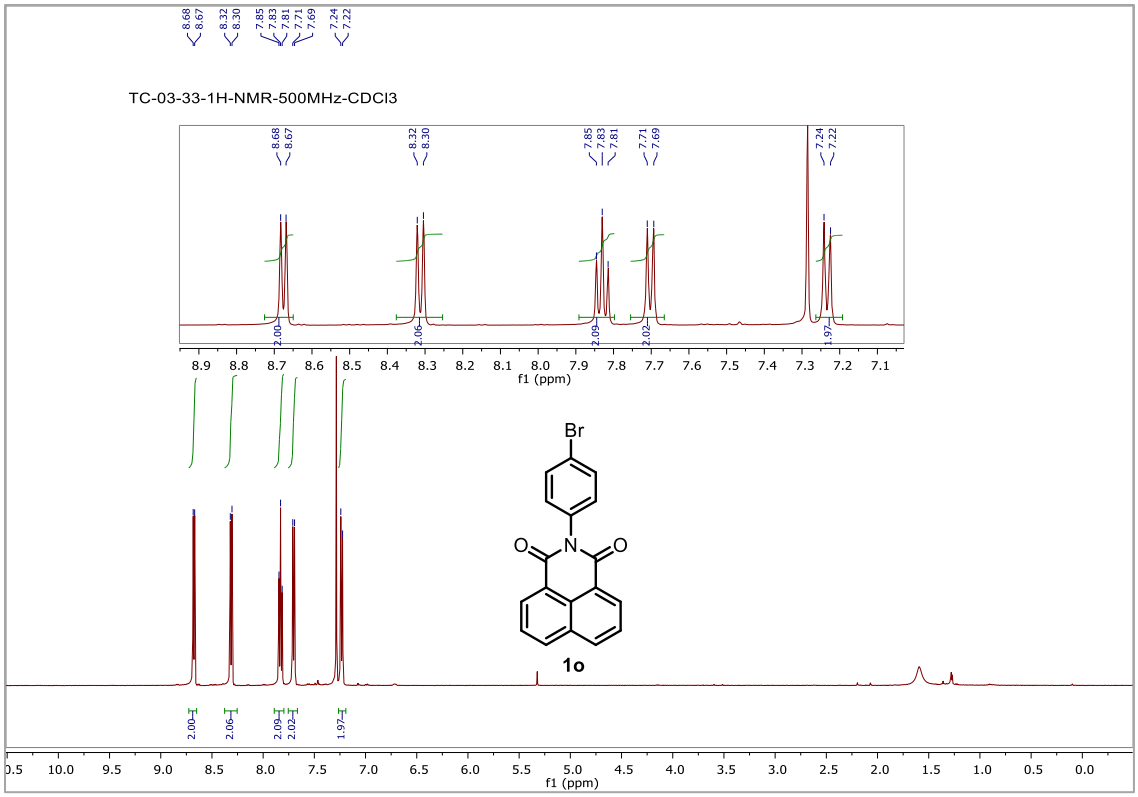
O
1n
f1 (ppm)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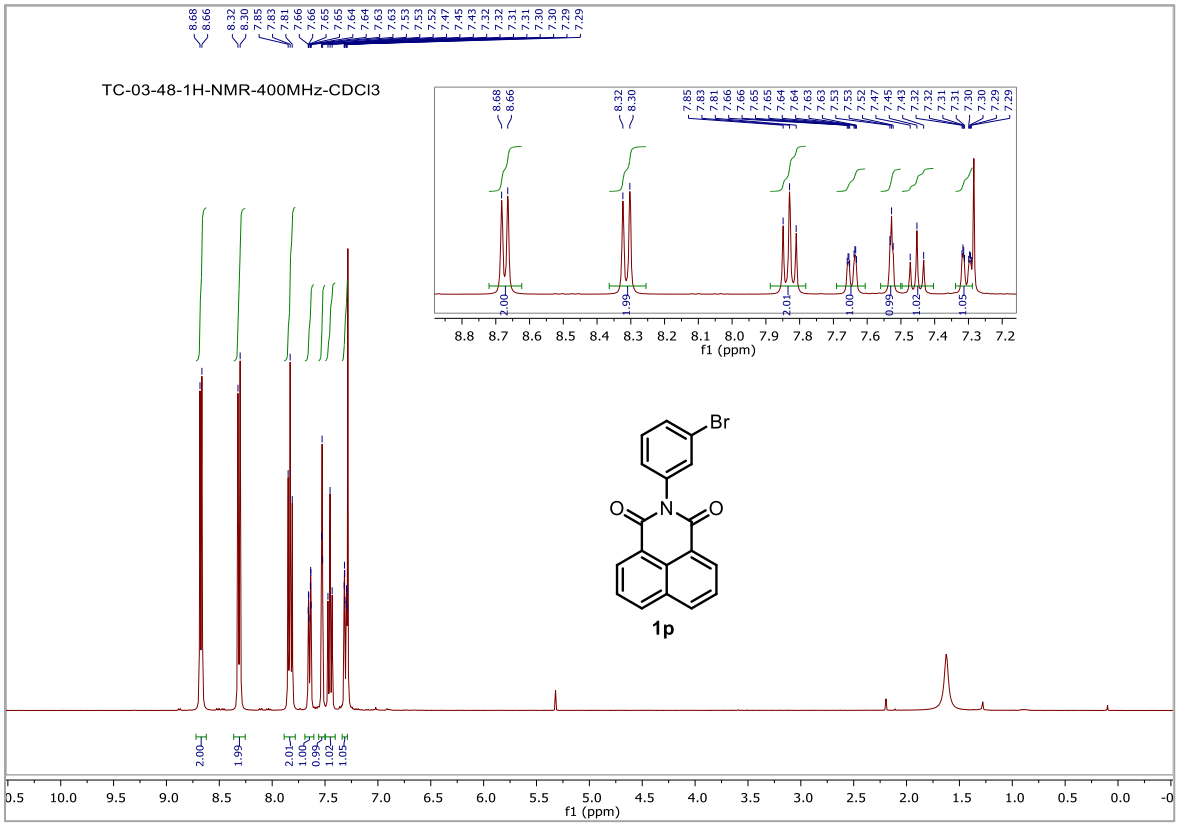
N
O
1o
f1 (ppm)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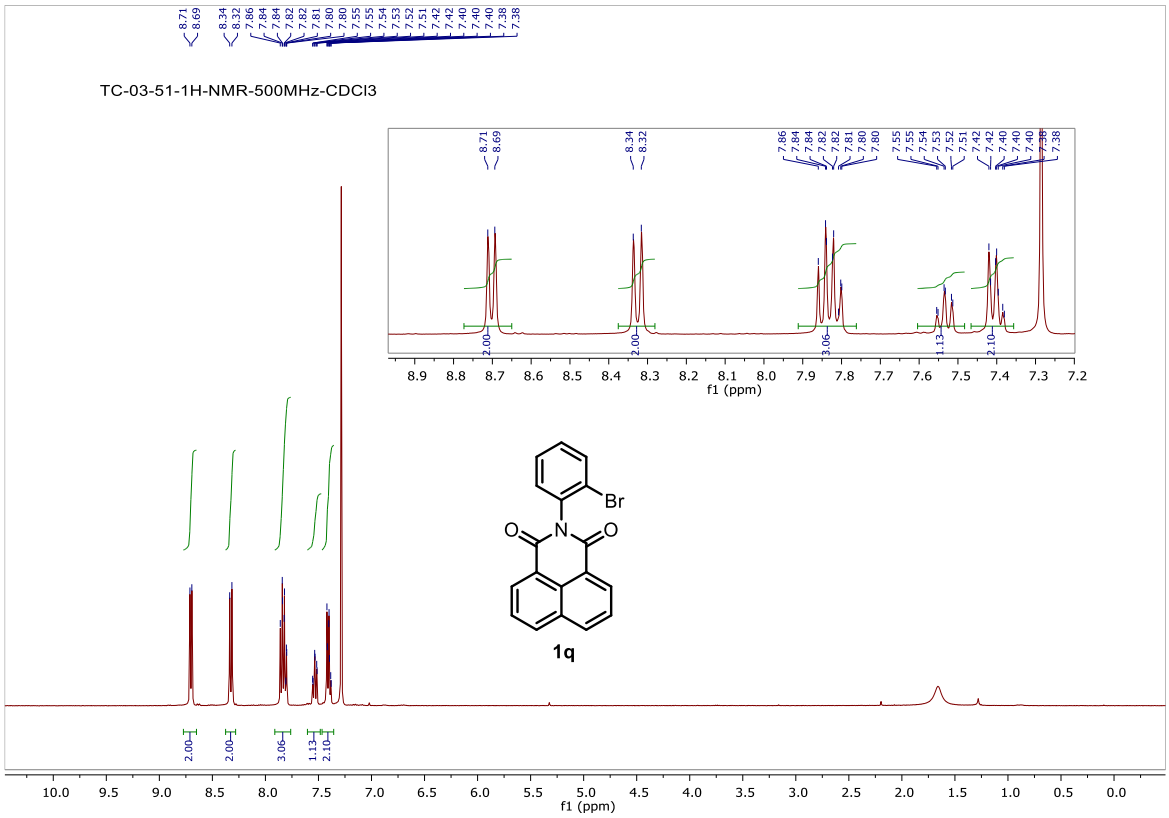
1p
f1 (ppm)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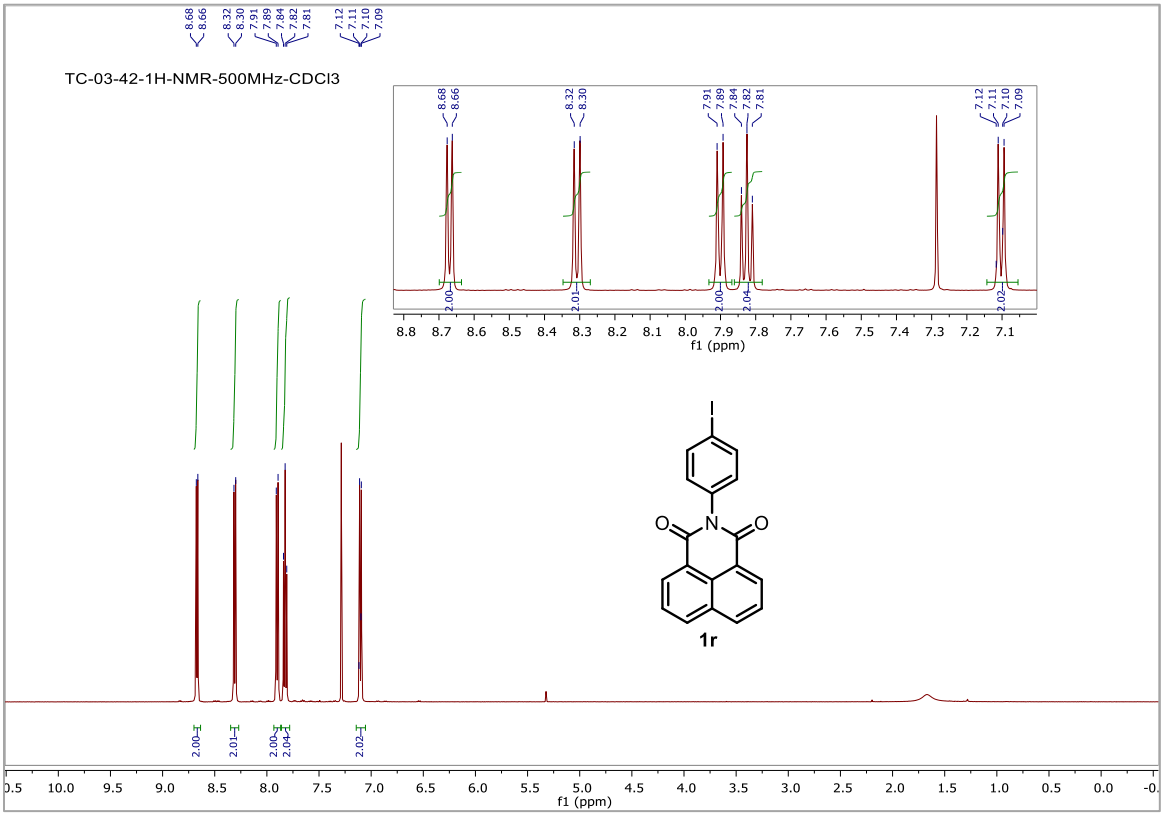
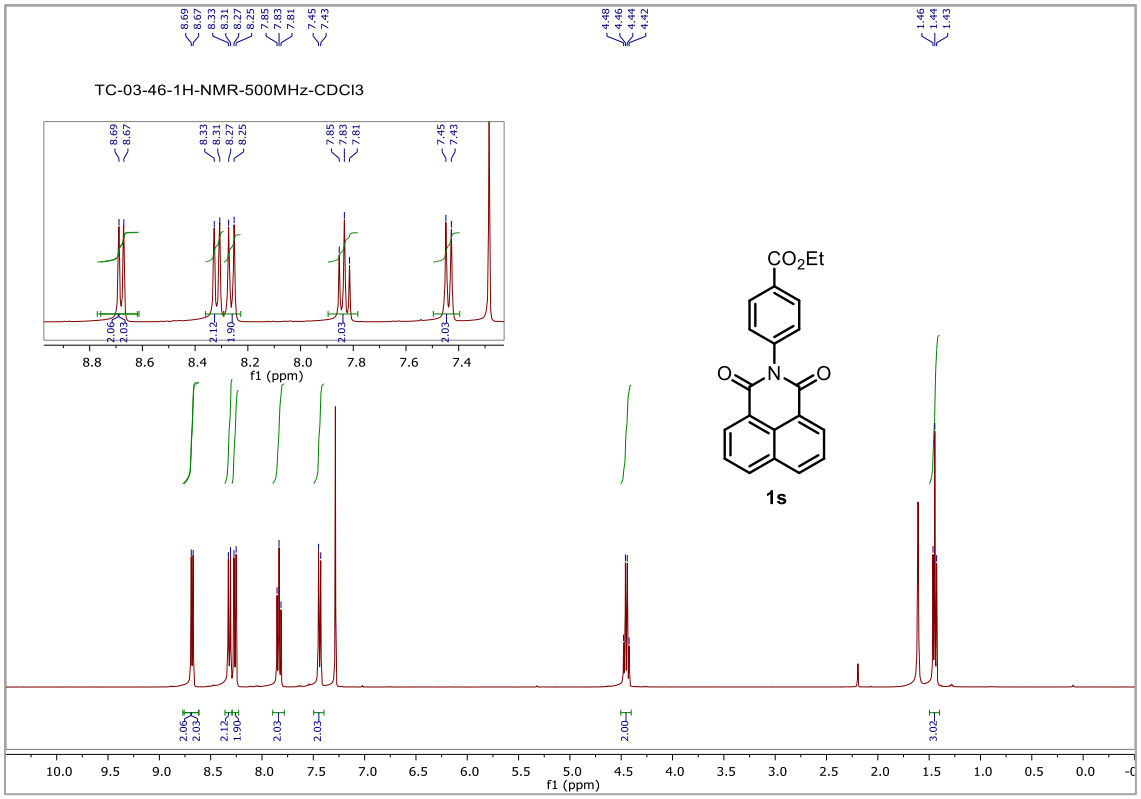
Br
O
N
O
1q
f1 (ppm)


TC-03-42-1H-NMR-500MHz-CDCl3
I
O
N
O
1r
f1 (ppm)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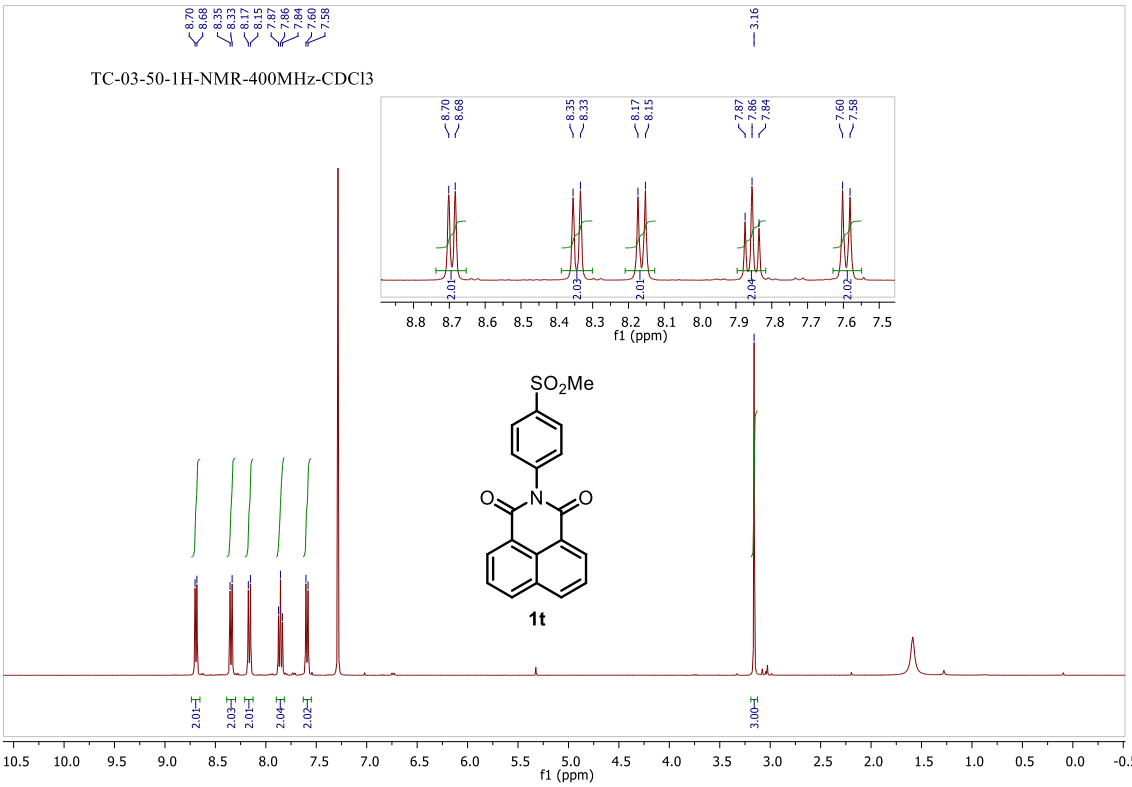
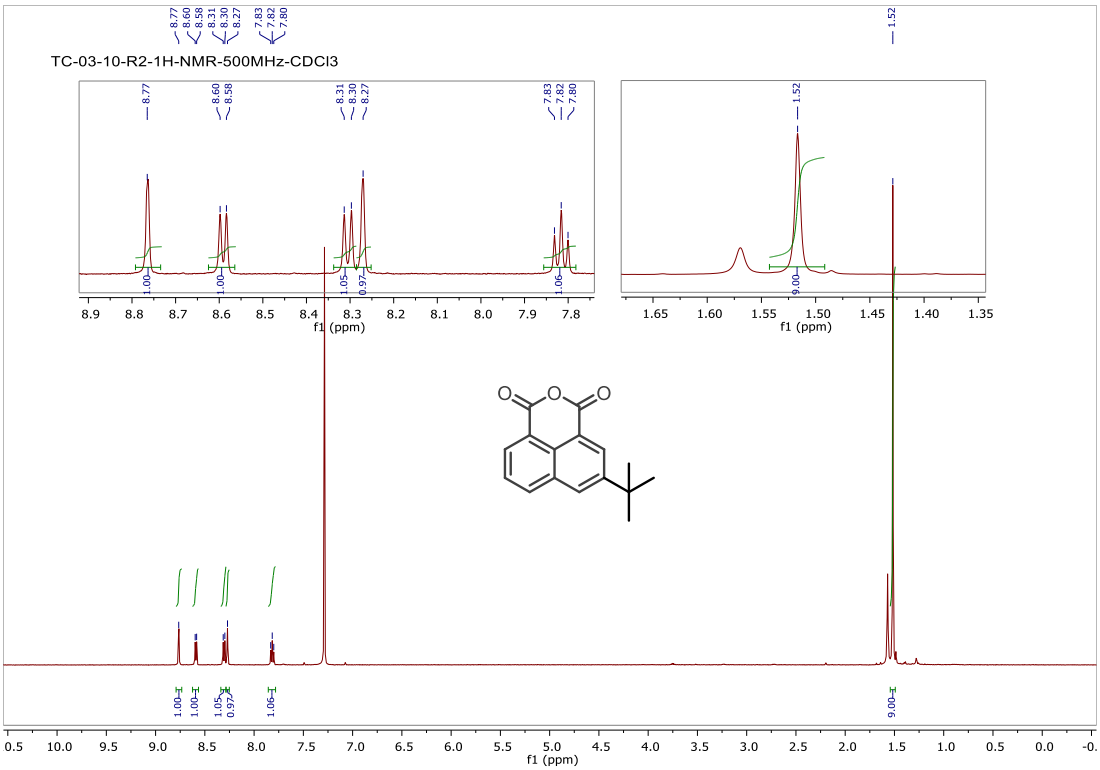
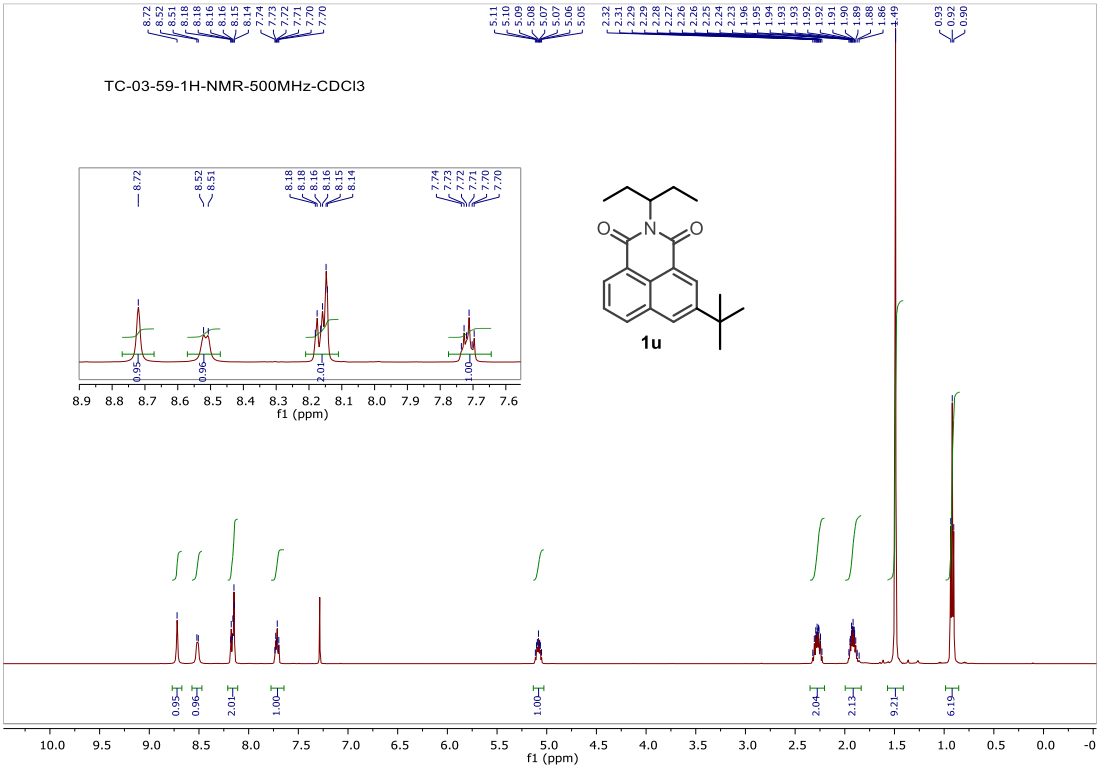
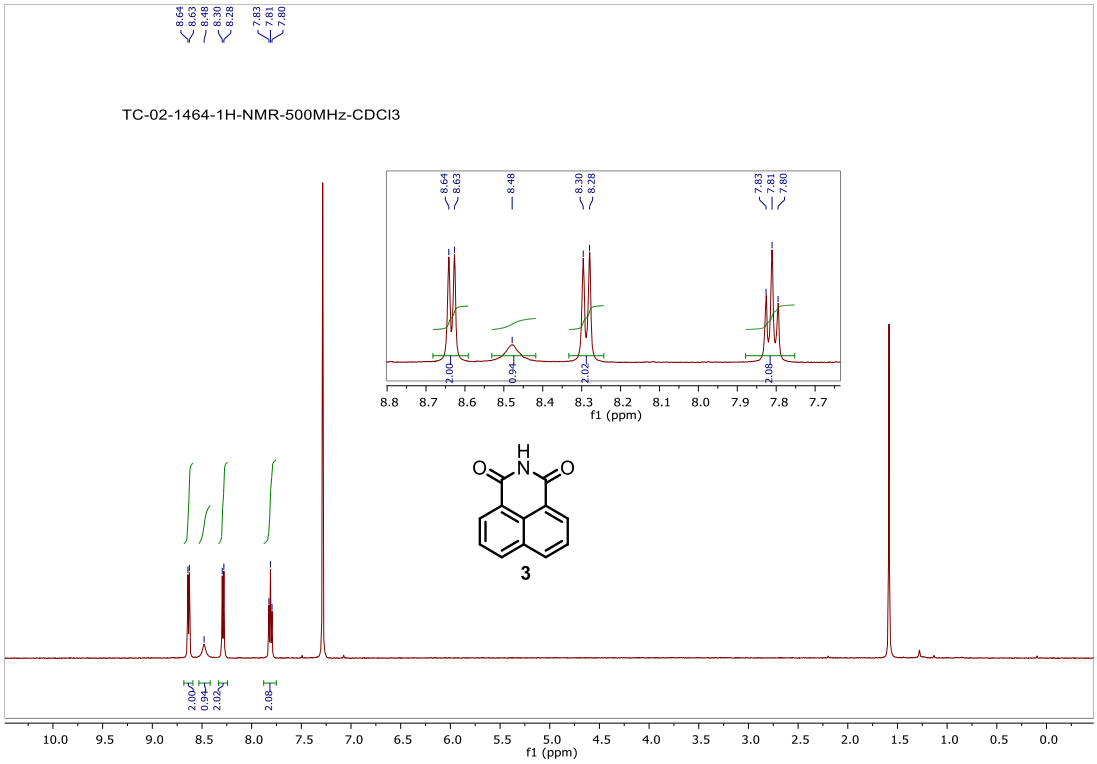
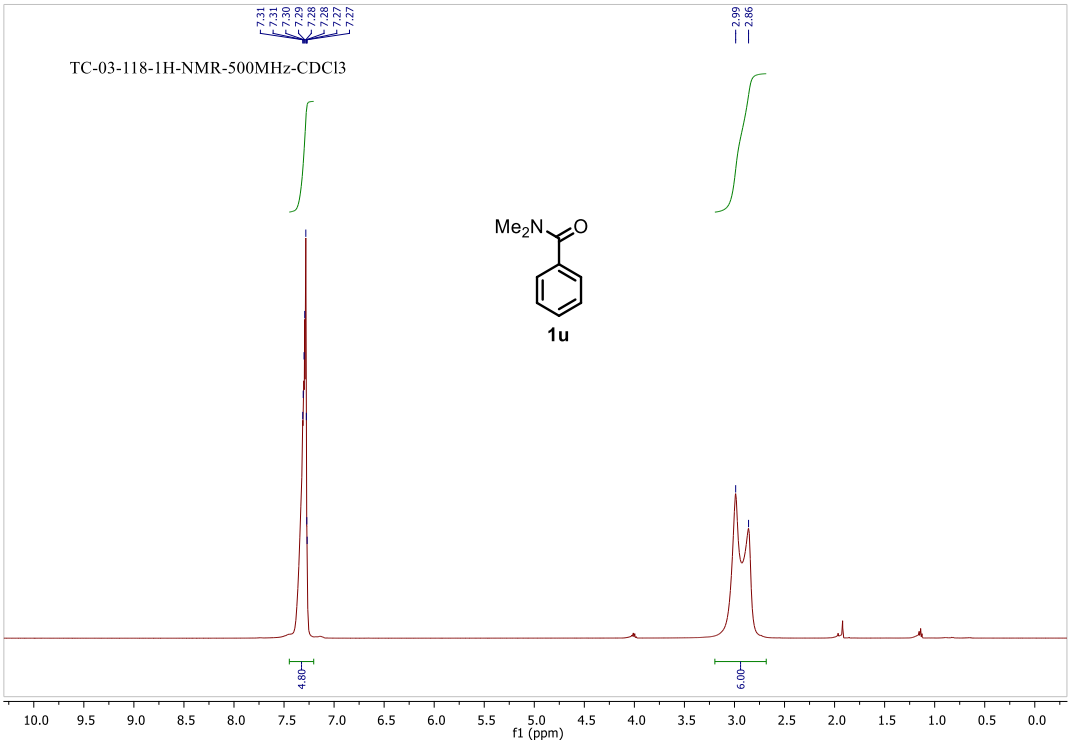
CO2Et
O
N
O
1s
f1 (ppm)

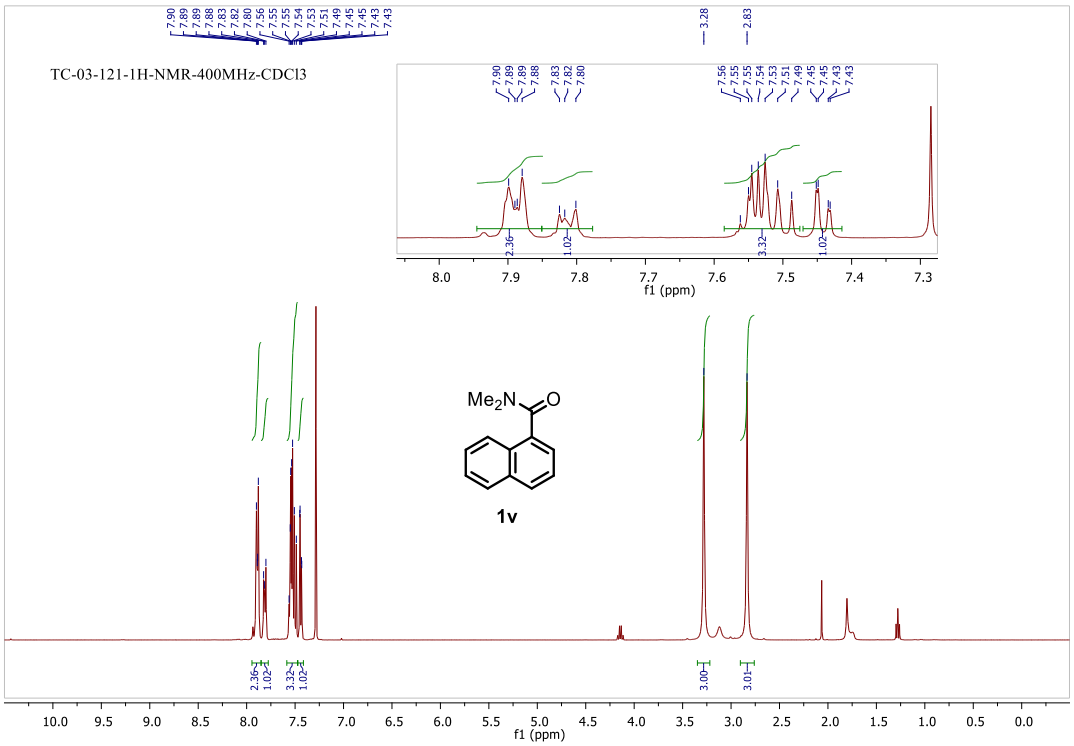

TC-03-50-1H-NMR-400MHz-CDCl3
SO2Me
O
N
O
1t
f1 (ppm)

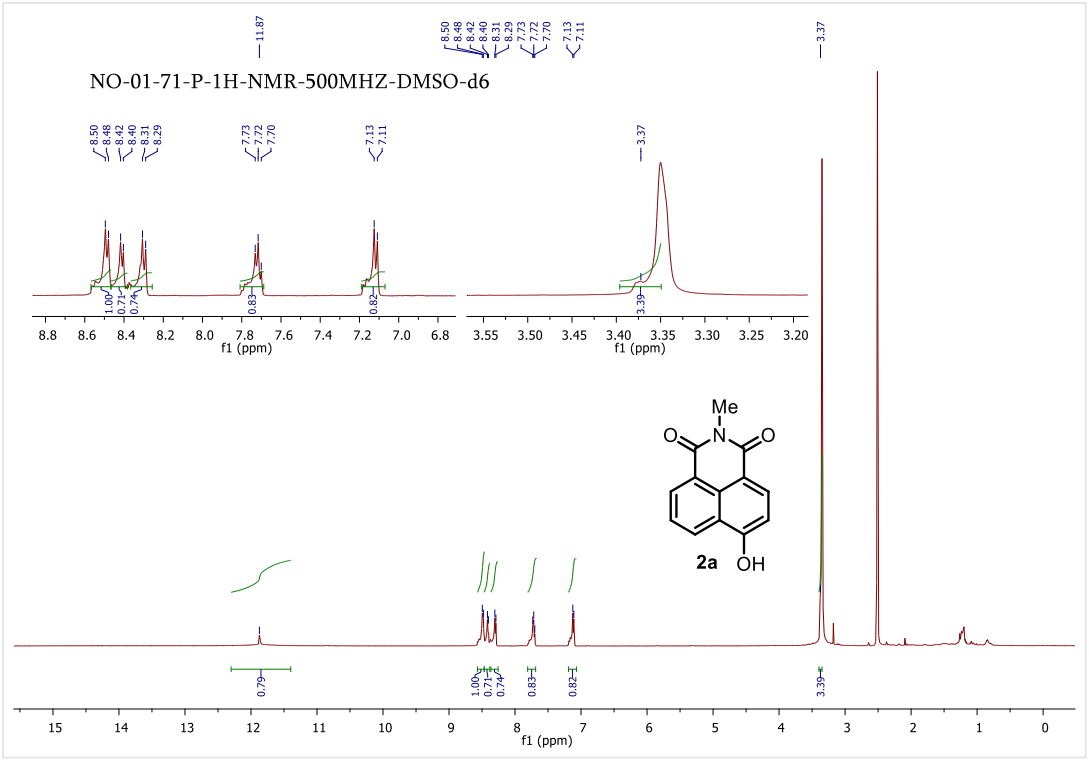

TC-03-10-R2-1H-NMR-500MHz-CDCl3
f1 (ppm)

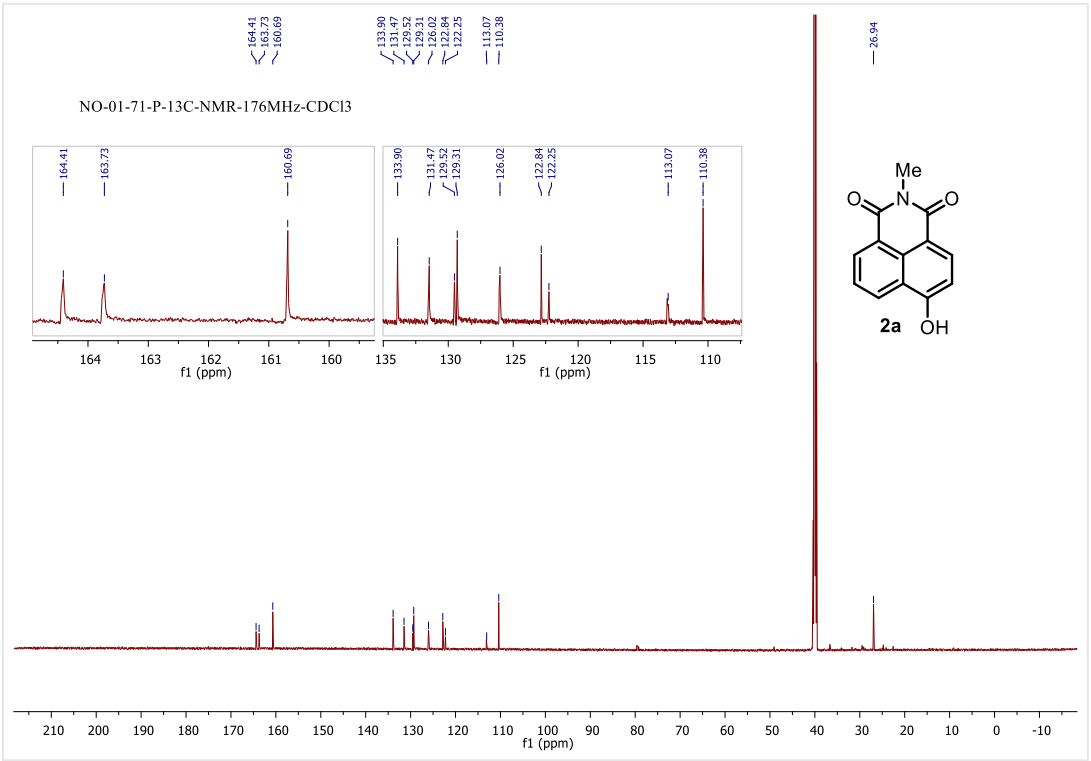

TC-03-59-1H-NMR-500MHz-CDCl3
1u
f1 (ppm)


TC-02-1464-1H-NMR-500MHz-CDCl3
8.64
8.63
8.48
8.30
8.28
7.83
7.81
7.80
2.00
0.94
2.02
2.08
f1 (ppm)
3


TC-03-118-1H-NMR-500MHz-CDCl3
7.31
7.30
7.29
7.28
7.27
2.99
2.86
4.80
6.00
f1 (ppm)
1u


TC-03-121-1H-NMR-400MHz-CDCl3
7.90
7.89
7.89
7.88
7.83
7.82
7.80
7.56
7.55
7.55
7.54
7.53
7.51
7.49
7.45
7.45
7.43
7.43
3.28
2.83
2.36
1.02
3.32
1.02
f1 (ppm)
Me2N
O
1v
2.36
1.02
3.32
1.02
3.00
3.01
f1 (ppm)


NO-01-71-P-1H-NMR-500MHZ-DMSO-d6
Me
N
O
O
2a
OH
f1 (ppm)


NO-01-71-P-13C-NMR-176MHz-CDCl3
Me
N
O
O
2a
OH
f1 (ppm)

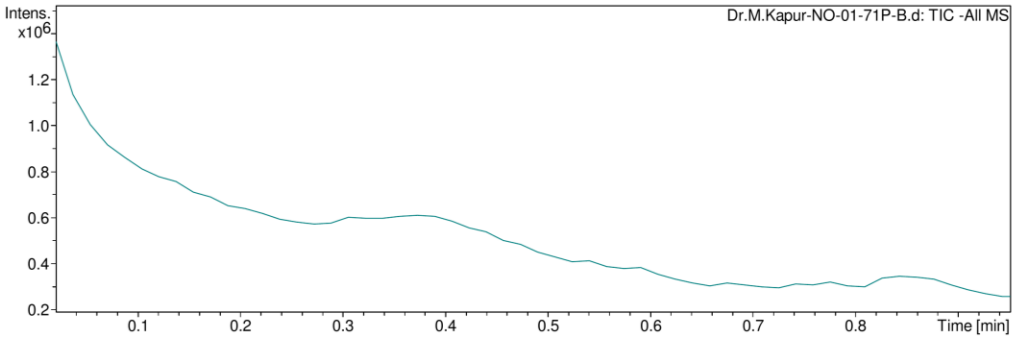
Display Report

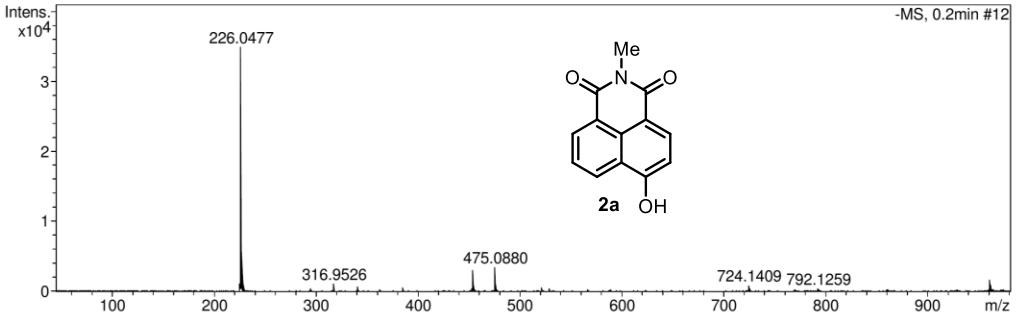
Analysis Info

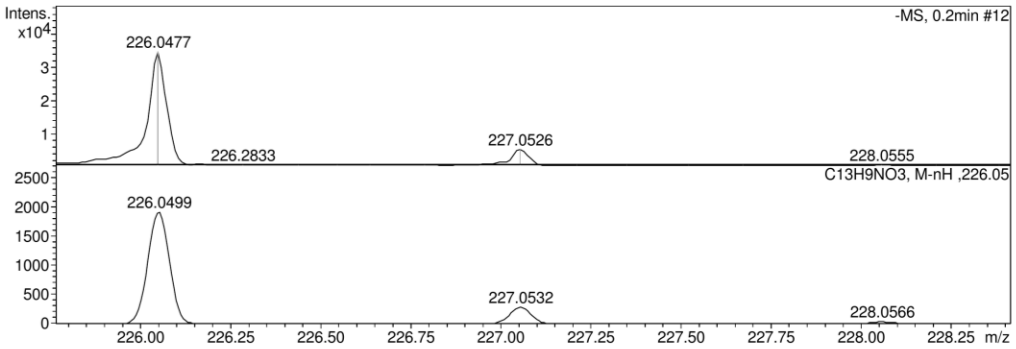
Acquisition Date	1/12/2022 4:17:35 PM		
Analysis Name	D:\Data\NEW USER DATA 2022\Jan-2022\12-jan-2022\Dr.M.Kapur-NO-01-71P-B.d		
Method	tune_low_neg.m	Operator	RUCHI
Sample Name	NO-01-71P-B	Instrument	micrOTOF-Q II 10330
Comment			

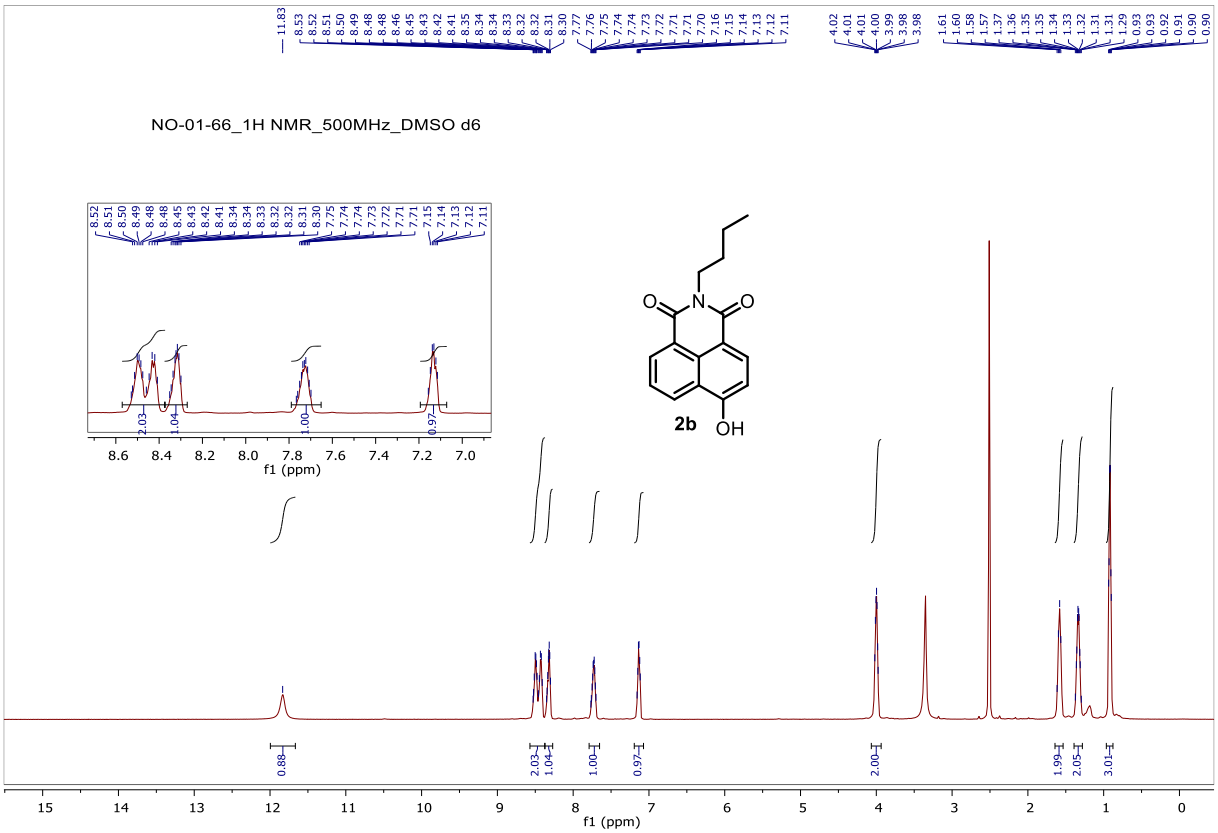
Acquisition Parameter

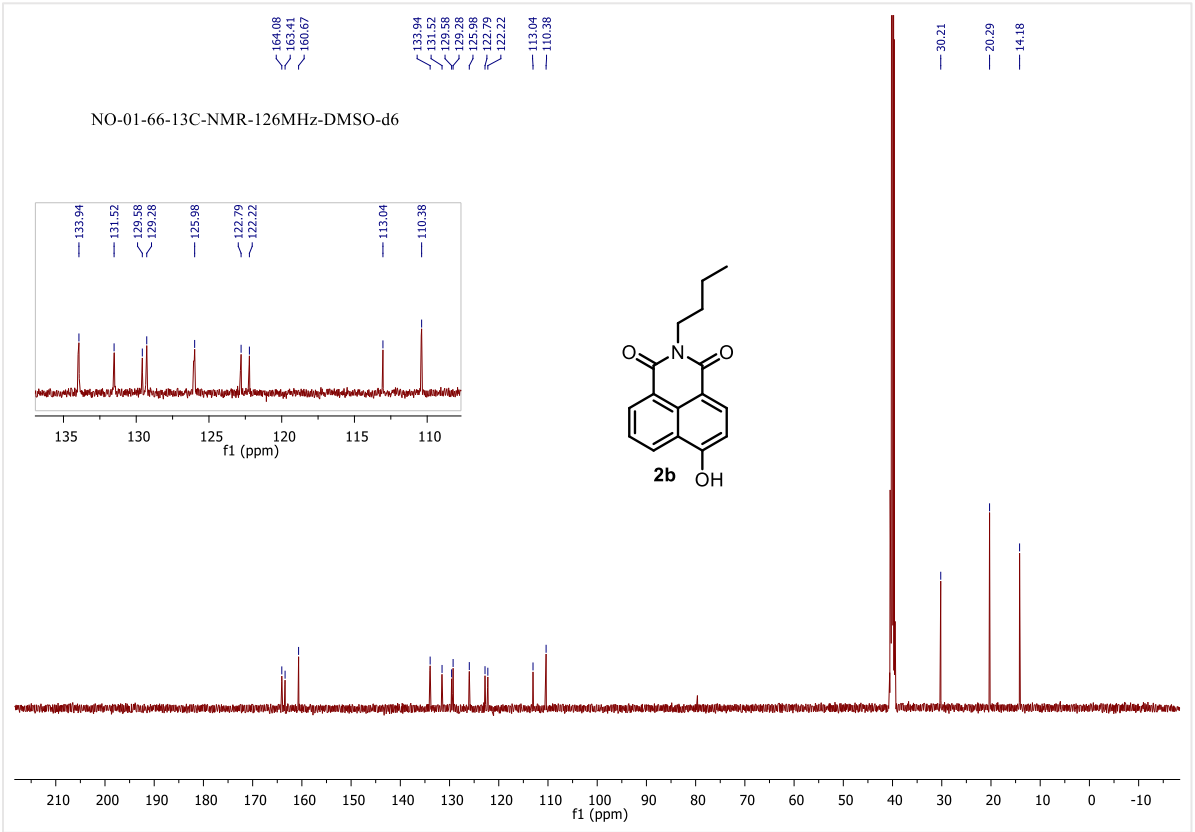
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Source








NO-01-66_1H NMR_500MHz_DMSO d6
2b
OH
f1 (ppm)


NO-01-66-13C-NMR-126MHz-DMSO-d6
2b
OH
f1 (ppm)

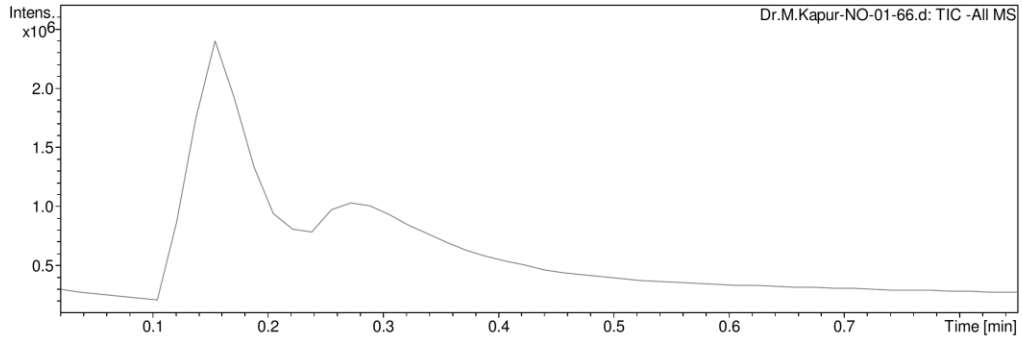
Display Report

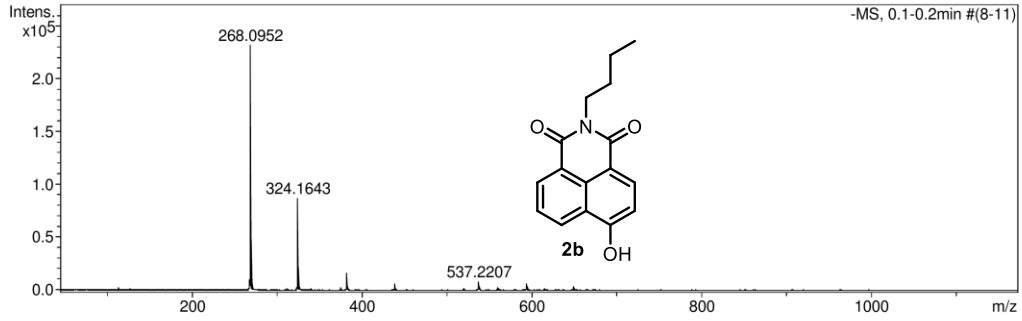
Analysis Info

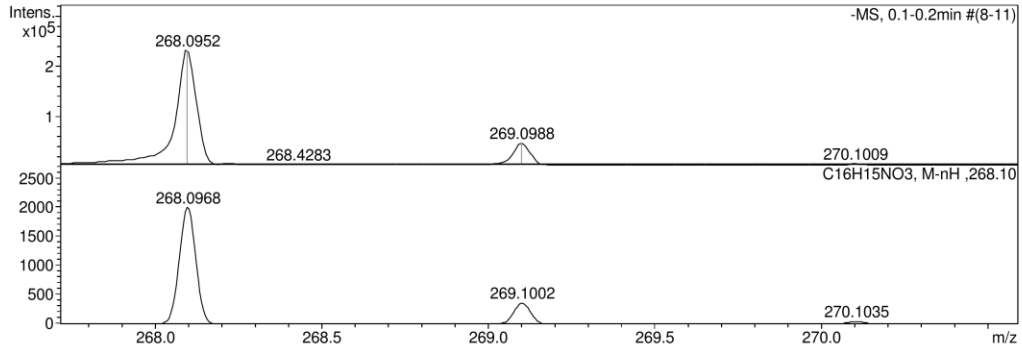
Analysis Name	D:\Data\NEW USER DATA 2022\Jan-2022\04-jan-2022\Dr.M.Kapur-NO-01-66.d	Acquisition Date	1/4/2022 4:01:56 PM
Method	tune_low_neg.m	Operator	RUCHI
Sample Name	NO-01-66	Instrument	micrOTOF-Q II 10330
Comment			

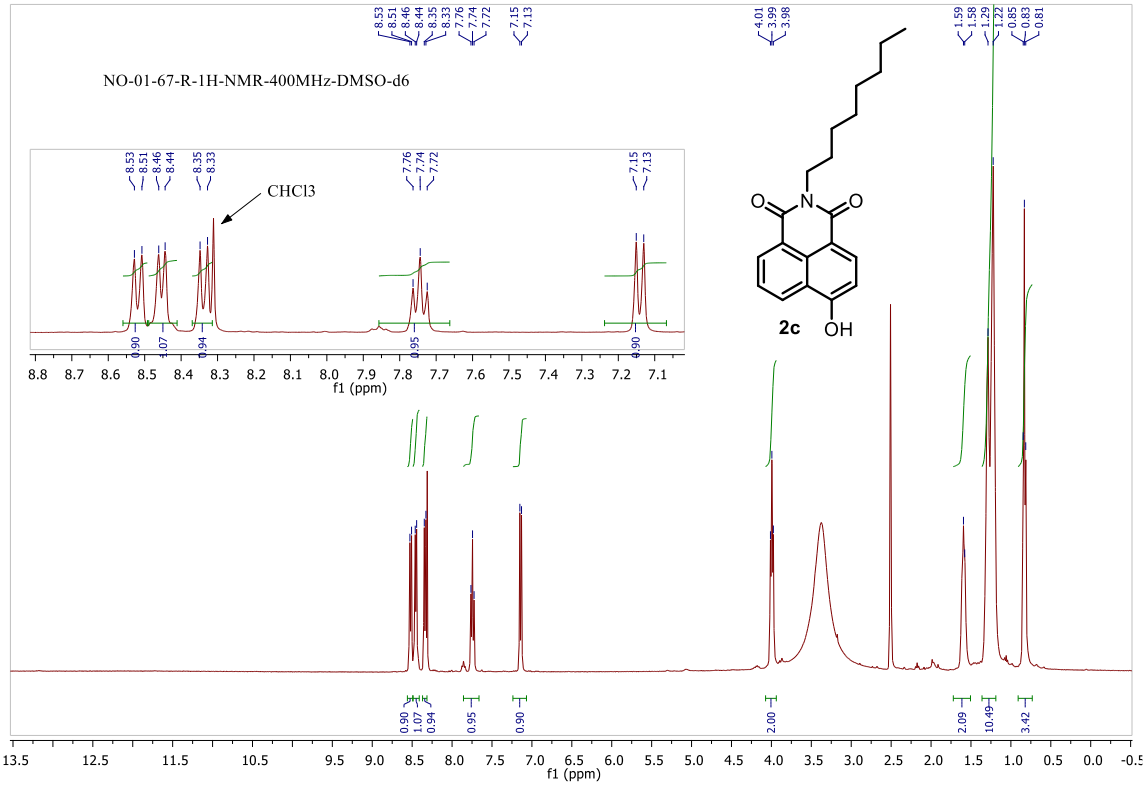
Acquisition Parameter

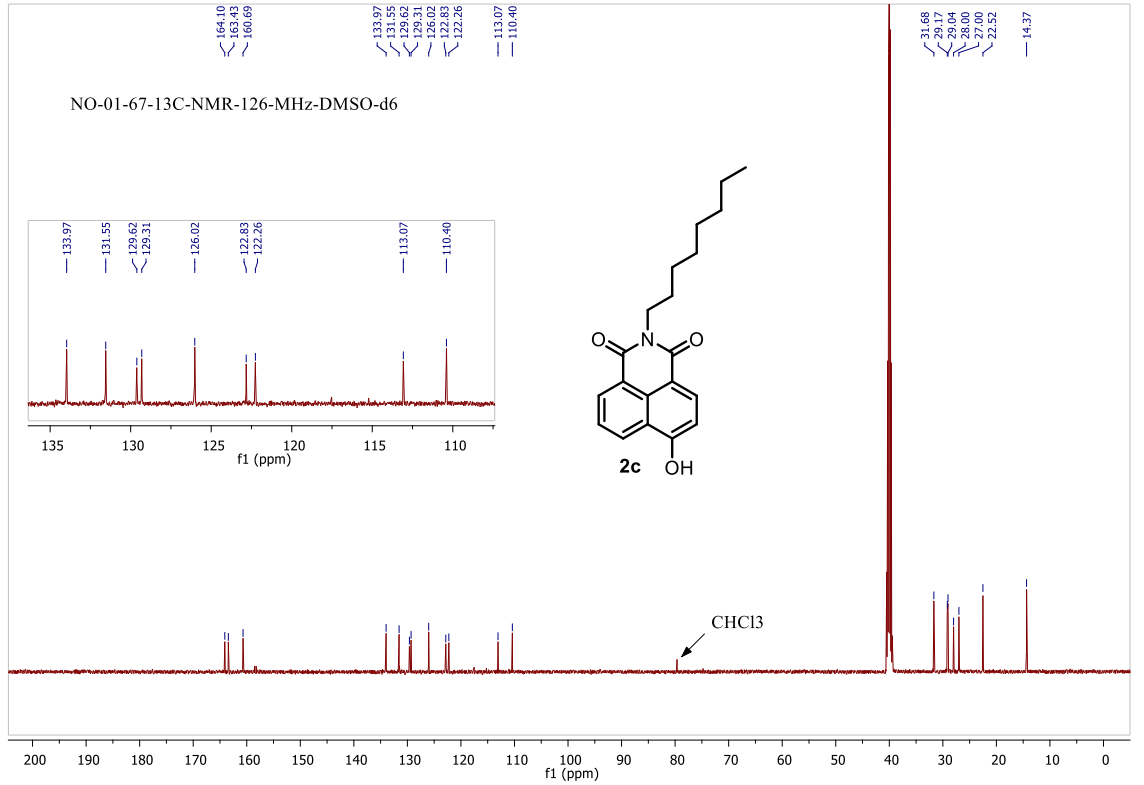
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste








NO-01-67-R-1H-NMR-400MHz-DMSO-d6
CHCl3
2c
OH
f1 (ppm)


NO-01-67-13C-NMR-126-MHz-DMSO-d6
2c
OH
CHCl3
f1 (ppm)

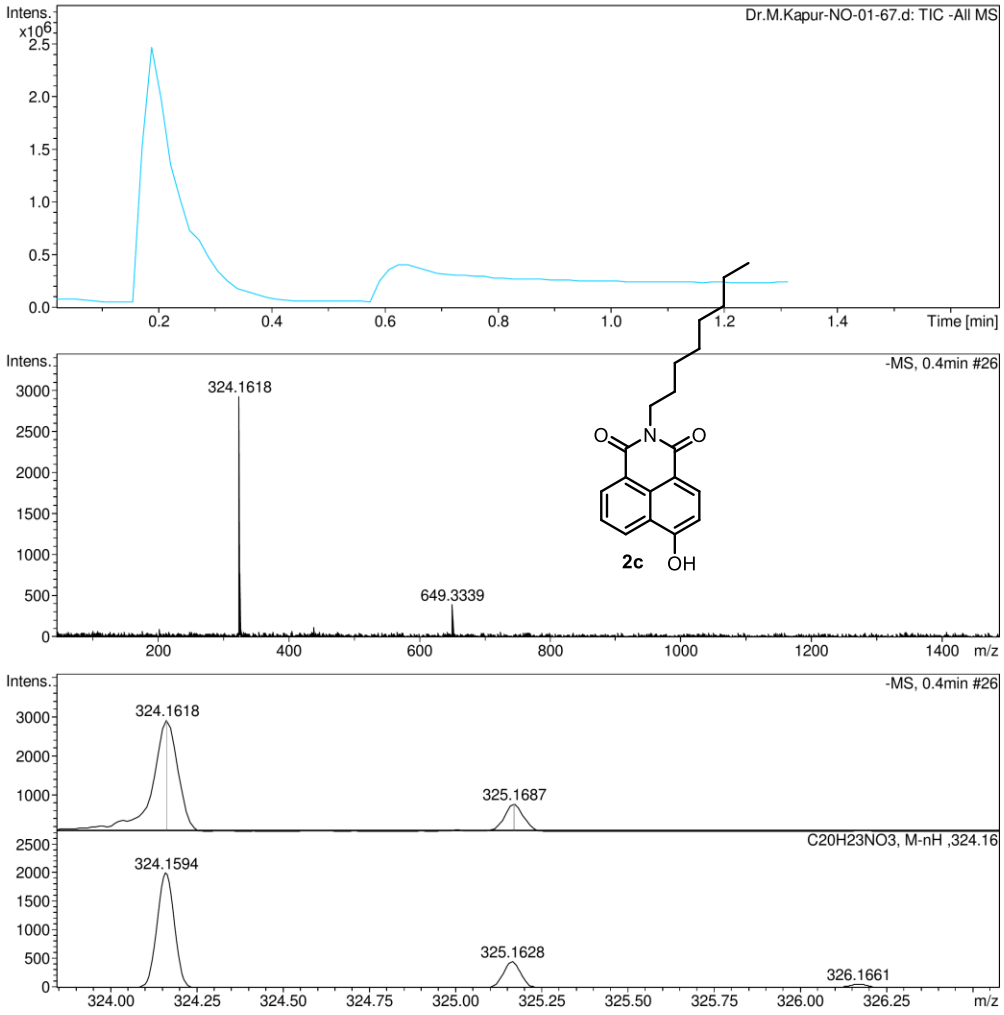
Display Report

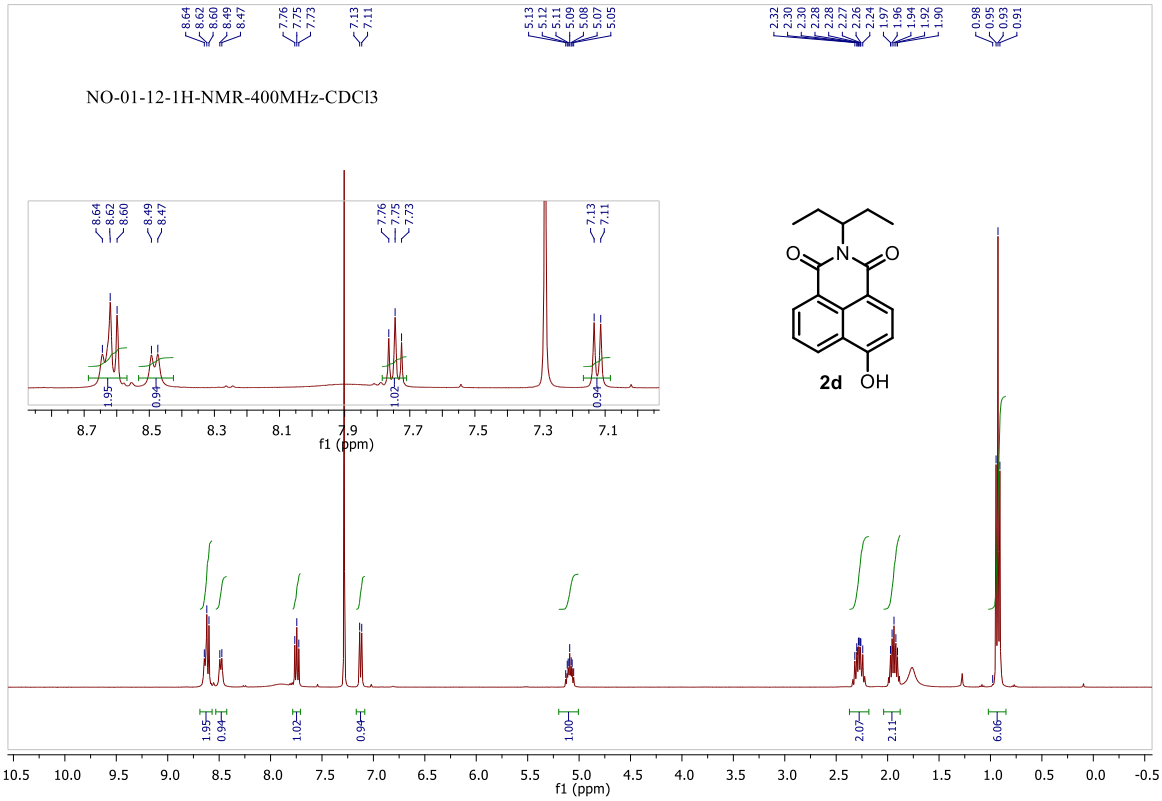
Analysis Info

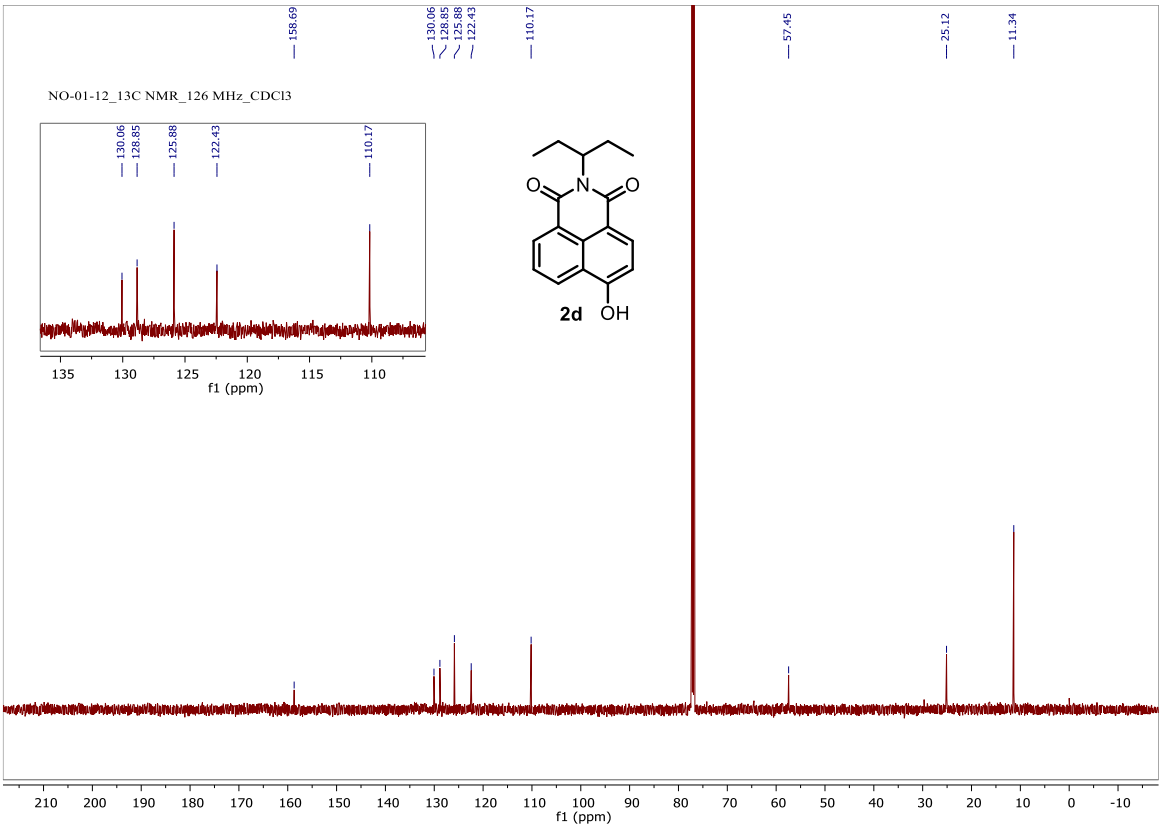
Analysis Name	D:\Data\NEW USER DATA 2022\Jan-2022\04-jan-2022\Dr.M.Kapur-NO-01-67.d	Acquisition Date	1/4/2022 3:59:04 PM
Method	tune_low_neg.m	Operator	RUCHI
Sample Name	NO-01-67	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste




NO-01-12-1H-NMR-400MHz-CDCl3
2d
f1 (ppm)


NO-01-12_13C NMR_126 MHz_CDCl3
2d
f1 (ppm)

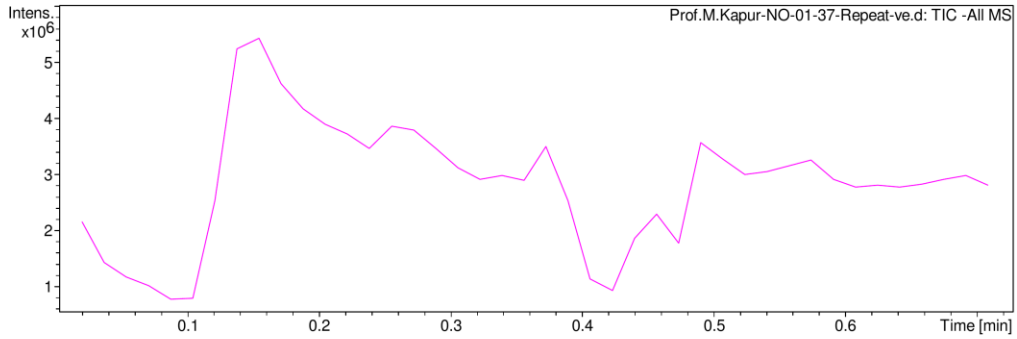
Display Report

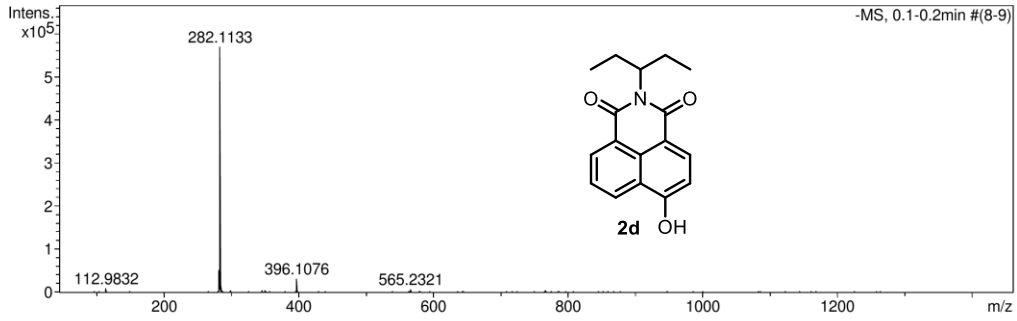
Analysis Info

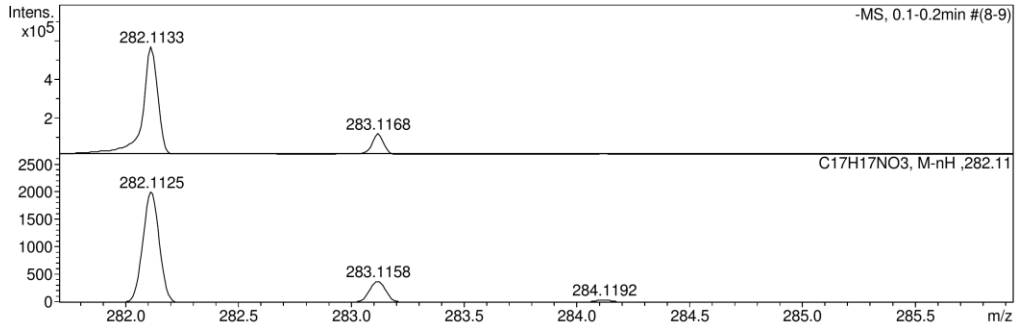
Acquisition Date	11/29/2021 11:37:20 AM		
Analysis Name	D:\Data\new user data 2021\Nov-2021\29-nov\Prof.M.Kapur-NO-01-37-Repeat-ve.d		
Method	tune mix_low.New.021117.m	Operator	RUCHI
Sample Name	NO-01-37-Repeat-ve	Instrument	micrOTOF-Q II 10330
Comment			

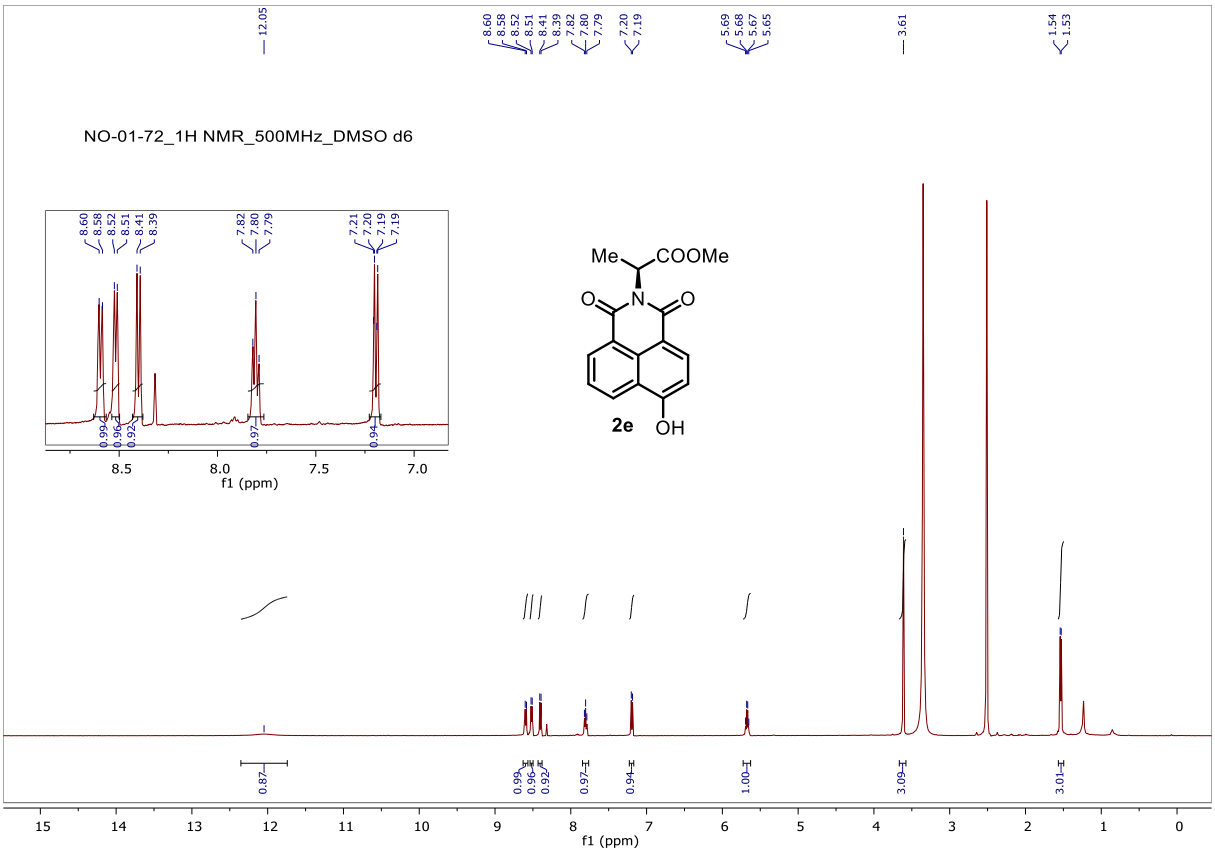
Acquisition Parameter

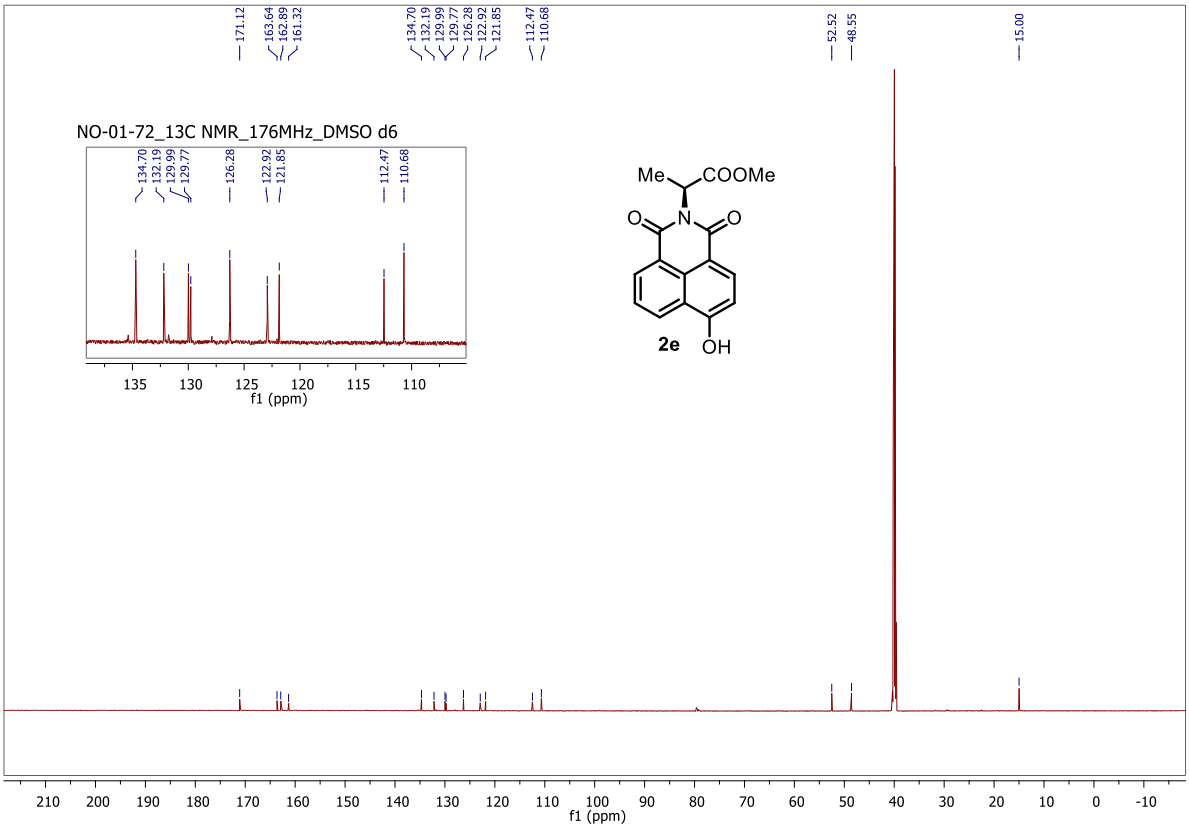
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Waste








NO-01-72_1H NMR_500MHz_DMSO d6
Me
COOMe
O
N
O
2e
OH
f1 (ppm)


NO-01-72_13C NMR_176MHz_DMSO d6
Me
COOMe
O
N
O
2e
OH
f1 (ppm)

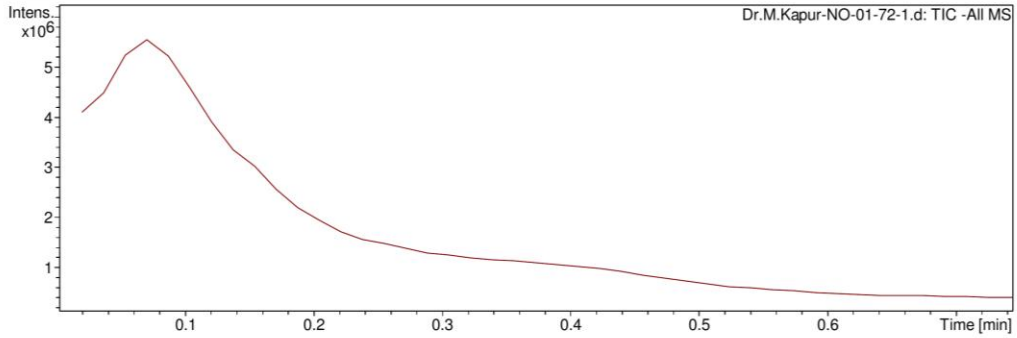
Display Report

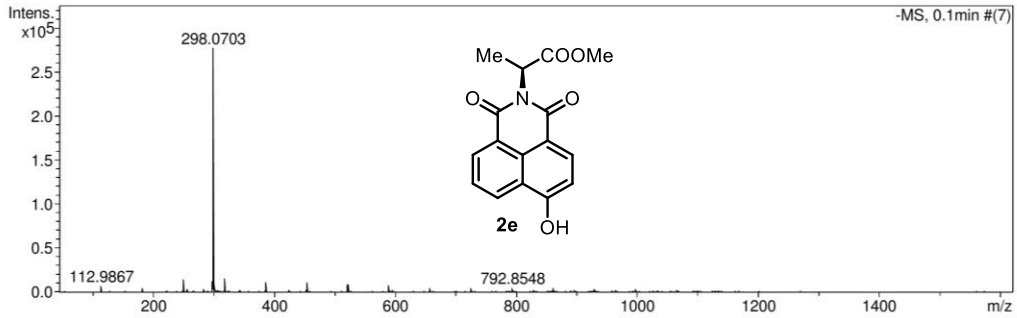
Analysis Info

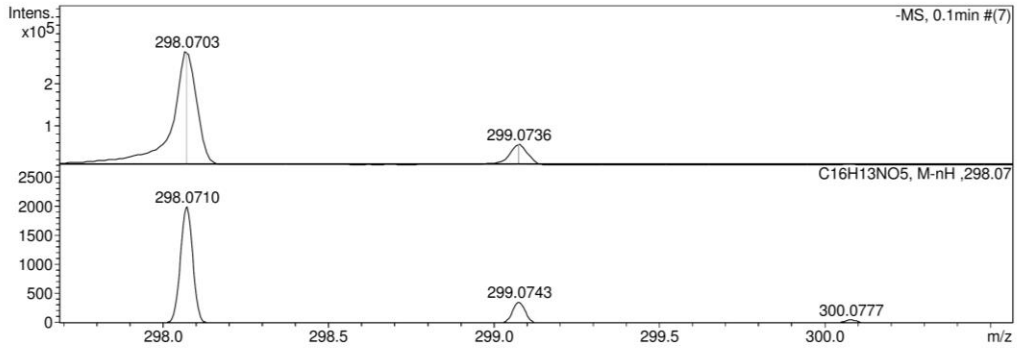
Acquisition Date	1/11/2022 4:13:08 PM		
Analysis Name	D:\Data\NEW USER DATA 2022\Jan-2022\11-JAN-2022\Dr.M.Kapur-NO-01-72-1.d		
Method	tune_low_neg.m	Operator	RUCHI
Sample Name	NO-01-72-1	Instrument	micrOTOF-Q II 10330
Comment			

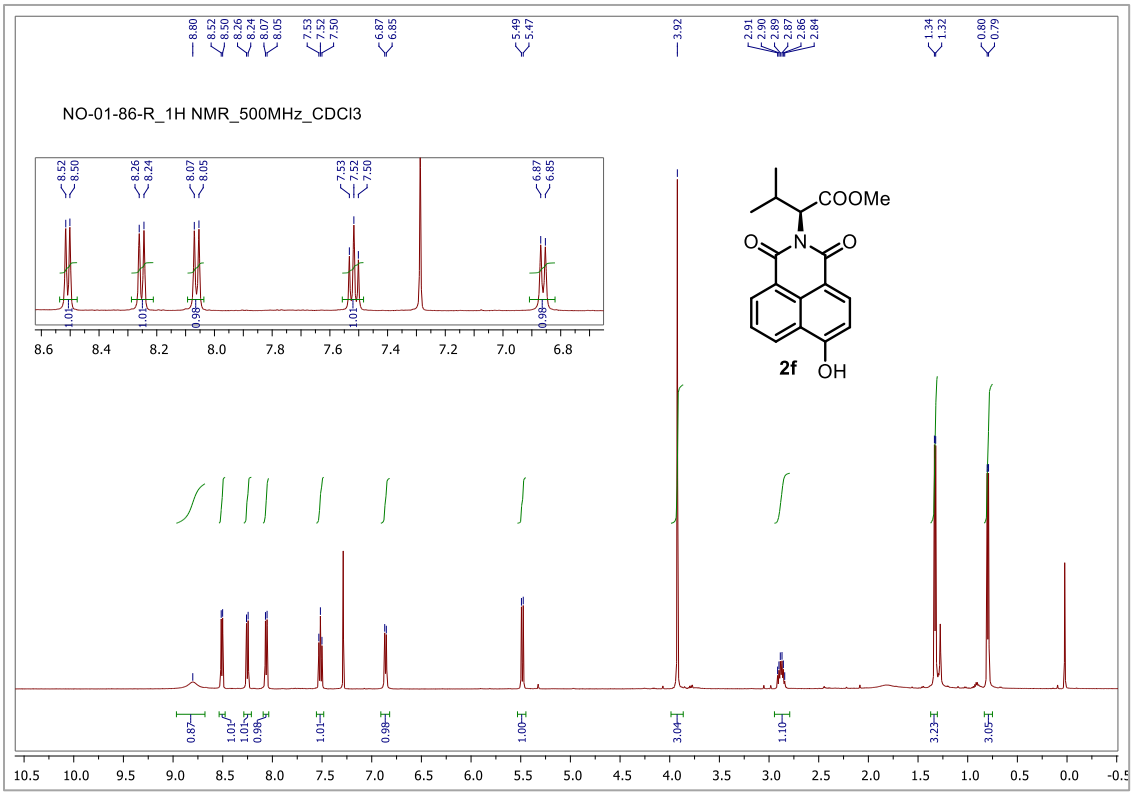
Acquisition Parameter

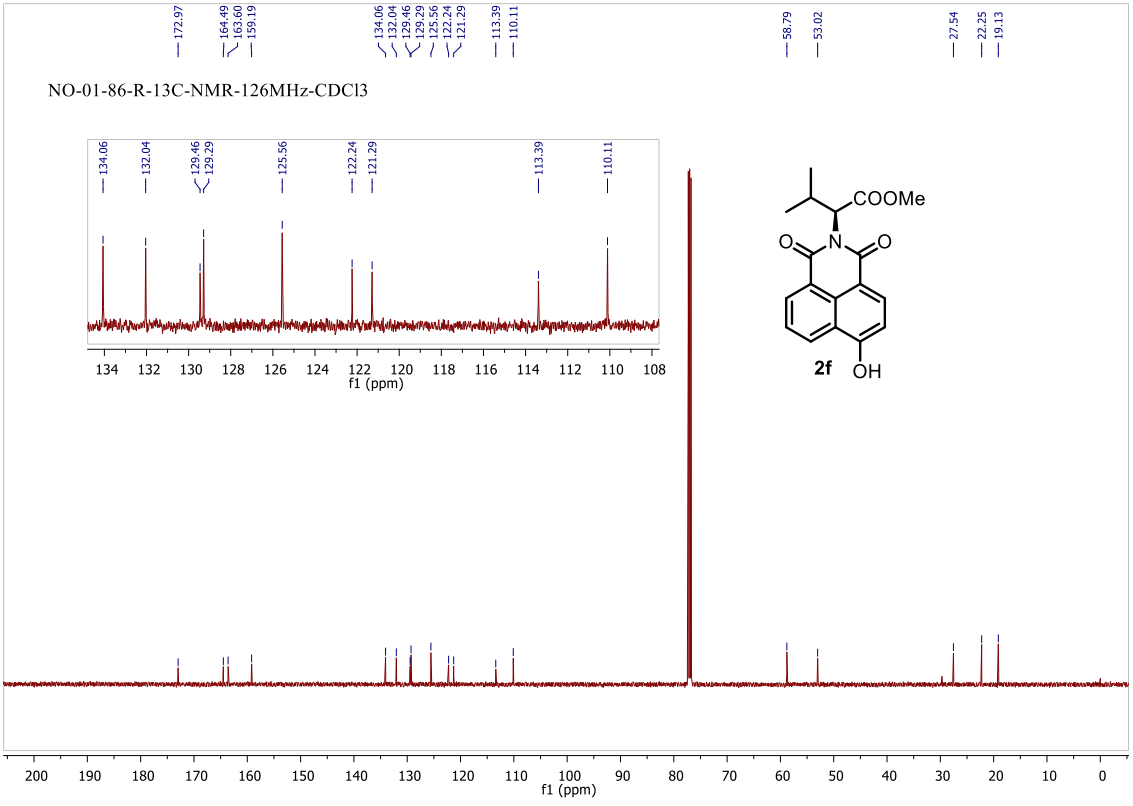
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Source








NO-01-86-R_1H NMR_500MHz_CDCl3
COOMe
2f
OH


NO-01-86-R-13C-NMR-126MHz-CDCl3
COOMe
2f
OH
f1 (ppm)

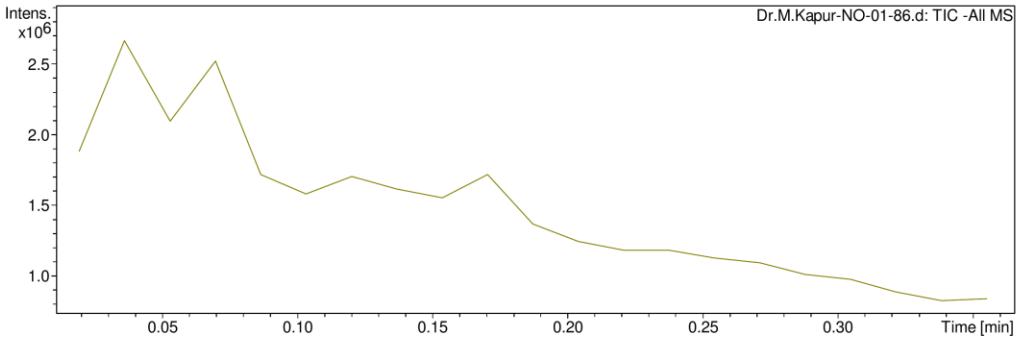
Display Report

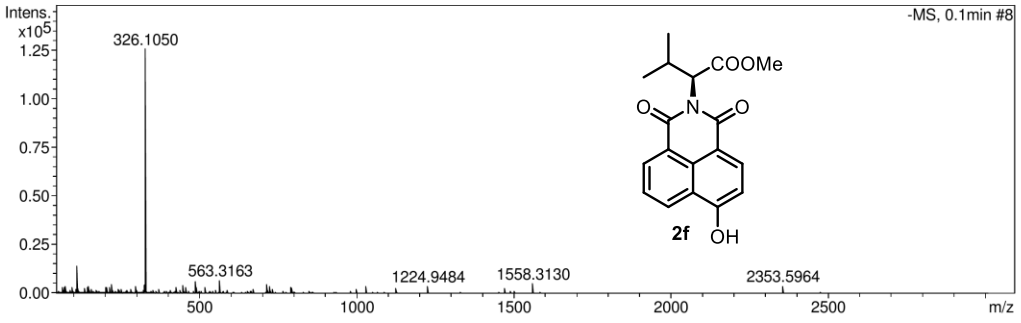
Analysis Info

Analysis Name	D:\Data\NEW USER DATA 2022\fe-2022\07-feb-2022\Dr.M.Kapur-NO-01-86.d	Acquisition Date	2/7/2022 4:28:38 PM
Method	tune mix_low.New.021117.m	Operator	RUCHI
Sample Name	NO-01-86	Instrument	micrOTOF-Q II 10330
Comment			

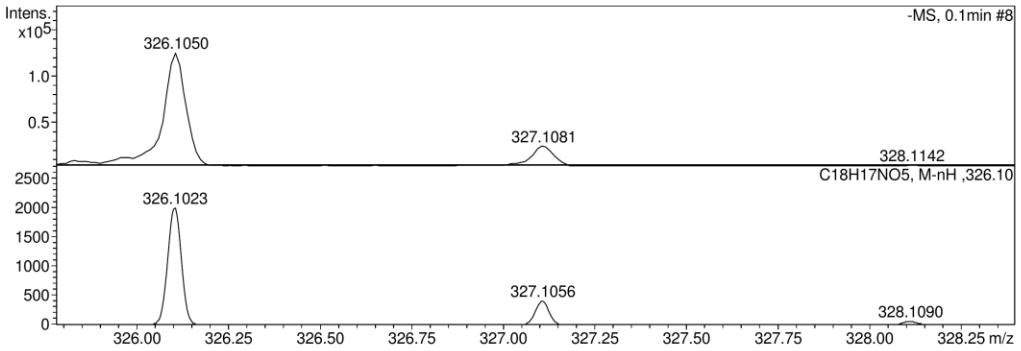
Acquisition Parameter

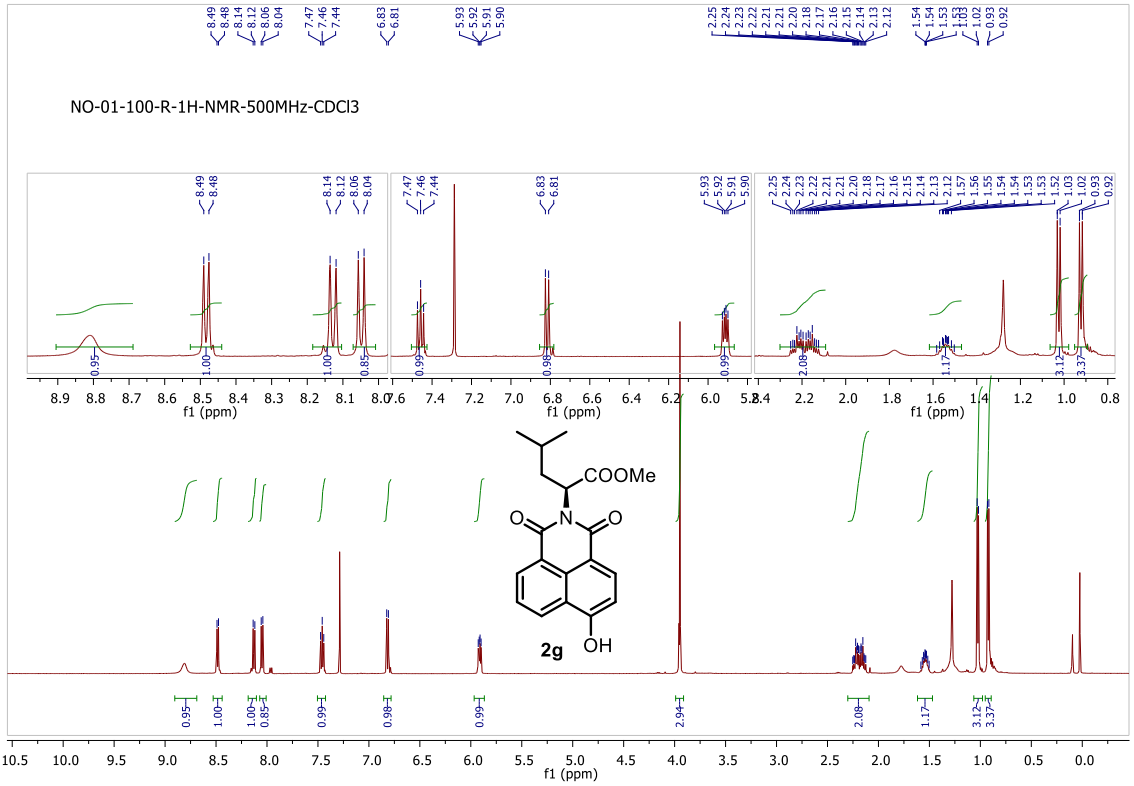
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Source

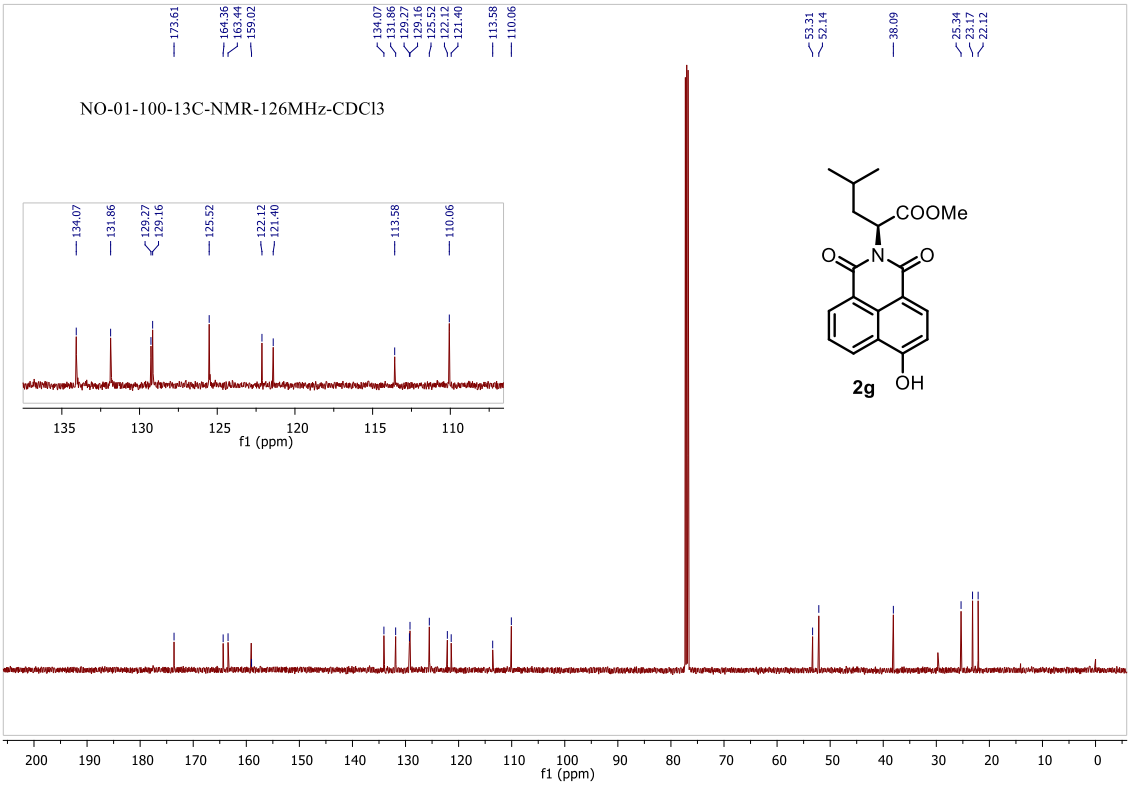




2f




NO-01-100-R-1H-NMR-500MHz-CDCl3
COOMe
2g
OH
f1 (ppm)


NO-01-100-13C-NMR-126MHz-CDCl3
COOMe
2g
OH
f1 (ppm)

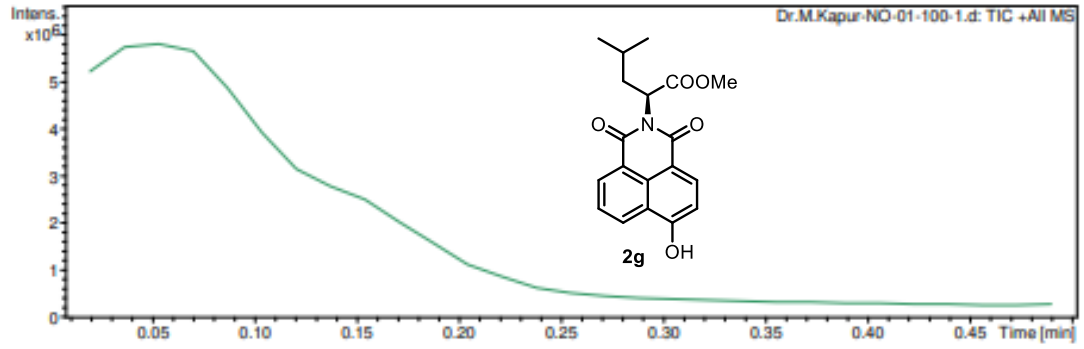
Display Report

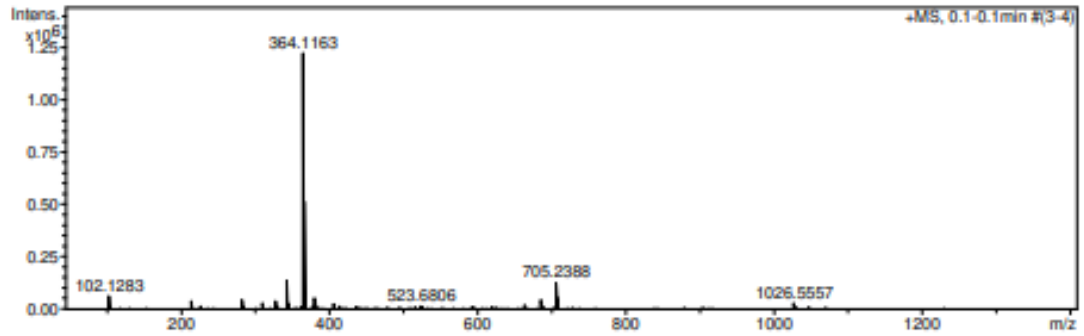
Analysis Info

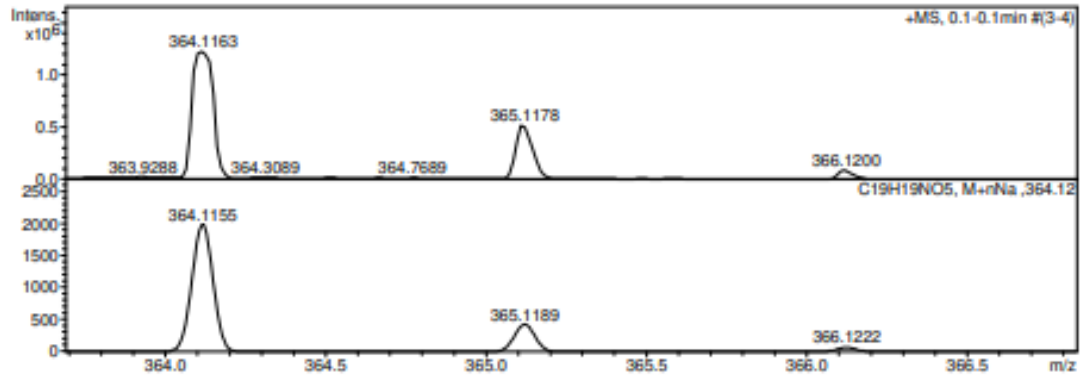
Analysis Name	D:\Data\NEW USER DATA 2022\April-2022\13-april\Dr.M.Kapur-NO-01-100-1.d	Acquisition Date	4/13/2022 3:13:45 PM
Method	tune mix_low.New.021117.m	Operator	RUCHI
Sample Name	NO-01-100-1	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

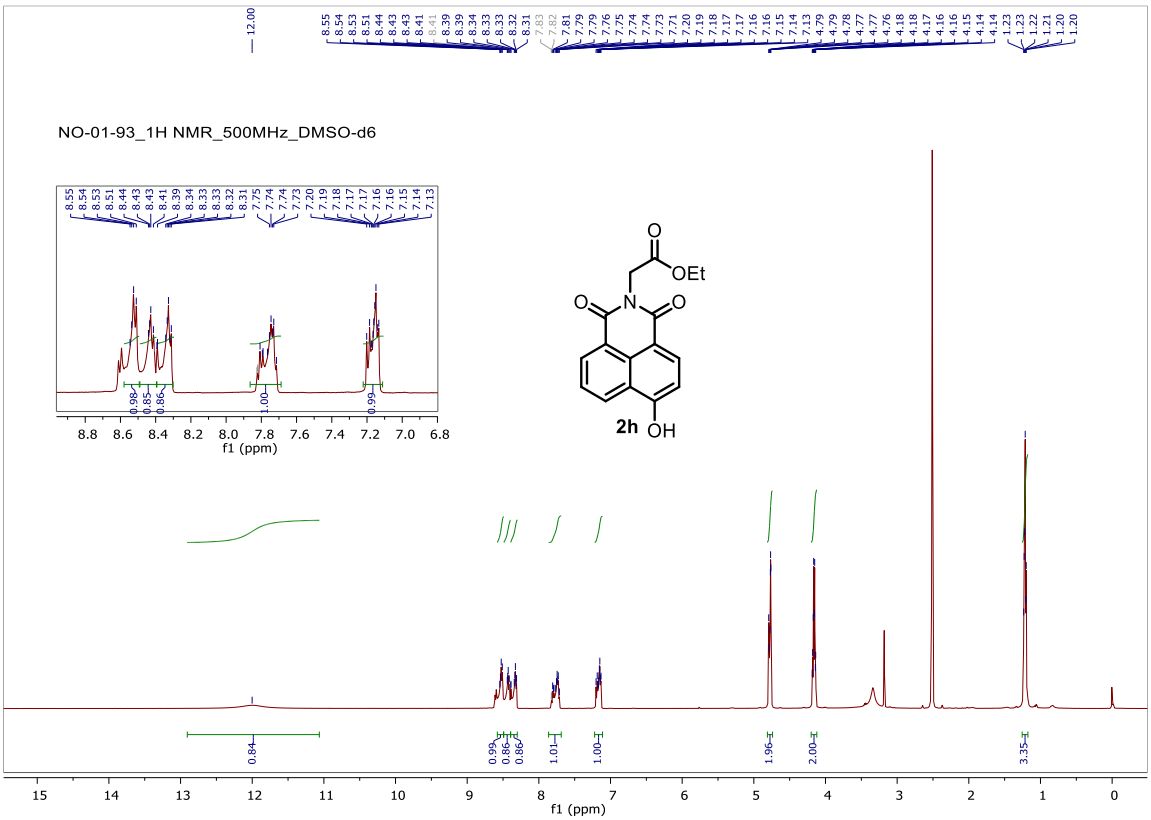
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Source

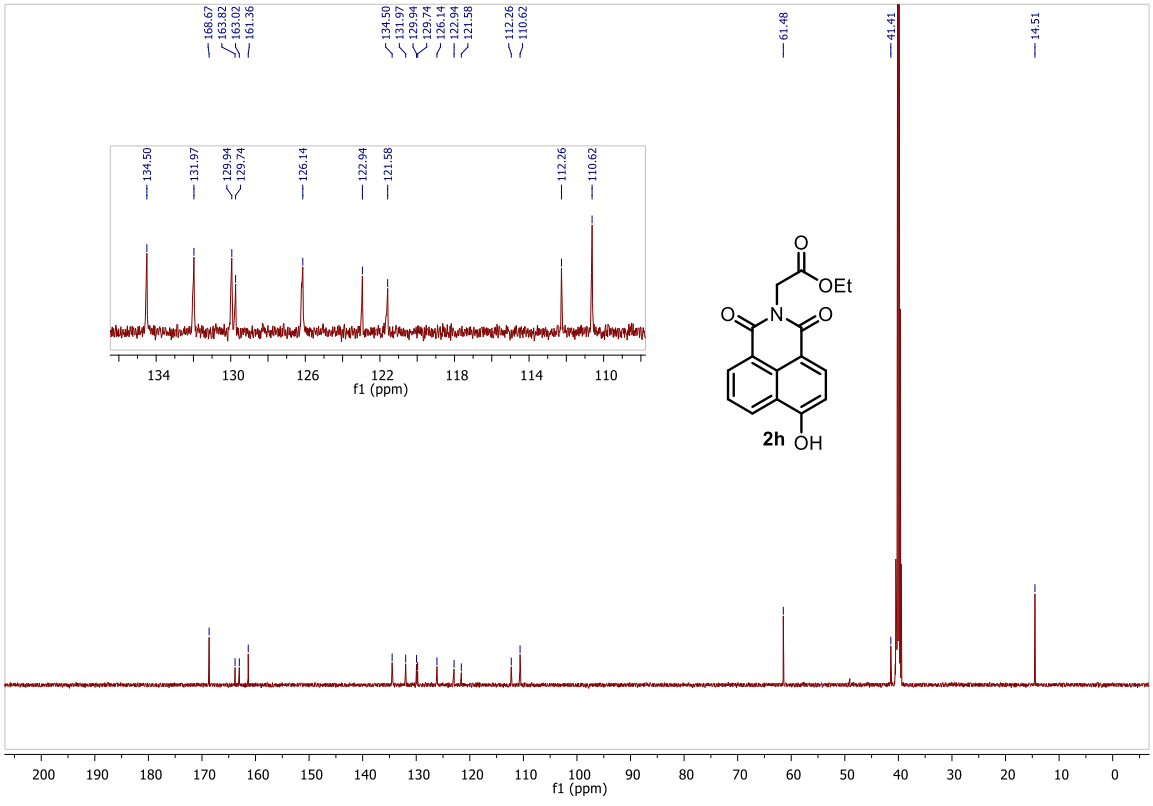






Bruker Compass DataAnalysis 4.0 printed: 4/13/2022 3:14:43 PM Page 1 of 1


NO-01-93_1H NMR_500MHz_DMSO-d6
f1 (ppm)
OEt
O
N
O
O
2h
OH


f1 (ppm)
OEt
O
N
O
O
2h
OH

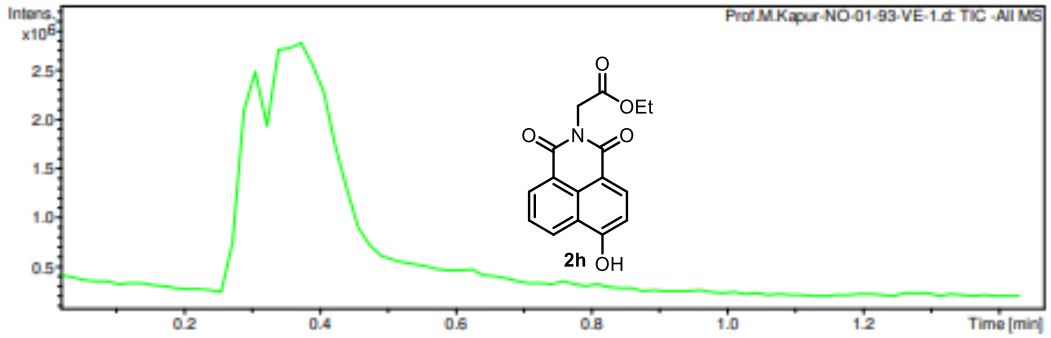
Display Report

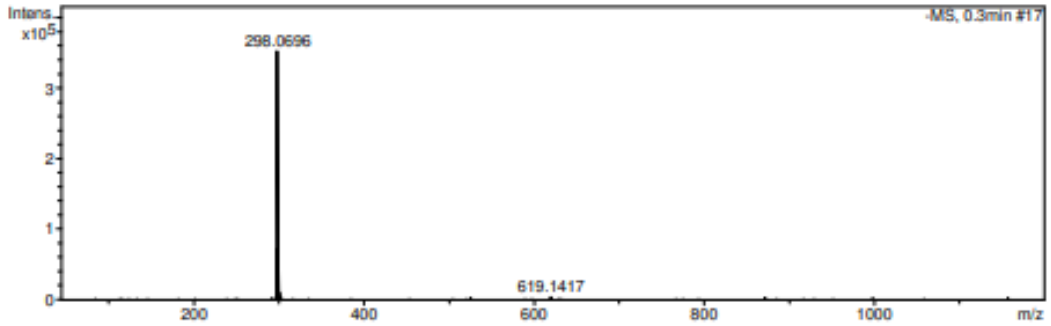
Analysis Info

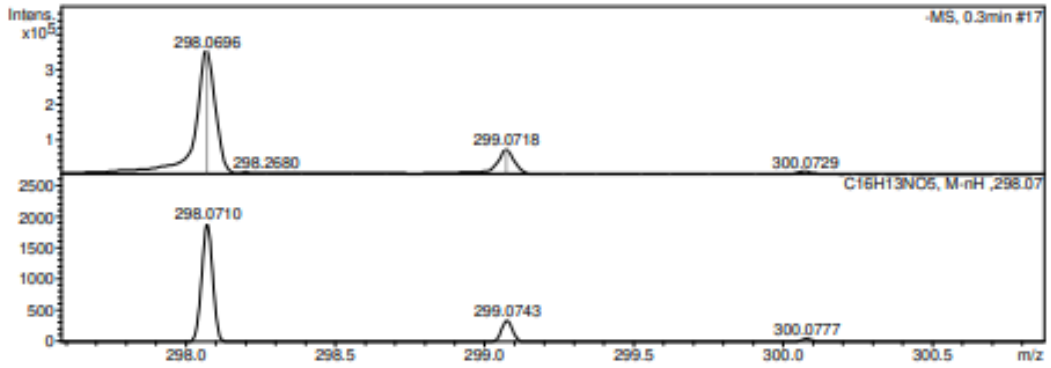
Acquisition Date	4/8/2022 2:23:00 PM		
Analysis Name	D:\Data\NEW USER DATA 2022\April-2022\08-april\Prof.M.Kapur-NO-01-93-VE-1.d		
Method	tune mix_low.New.021117.m	Operator	RUCHI
Sample Name	NO-01-93-VE-1	Instrument	micrOTOF-Q II 10330
Comment			

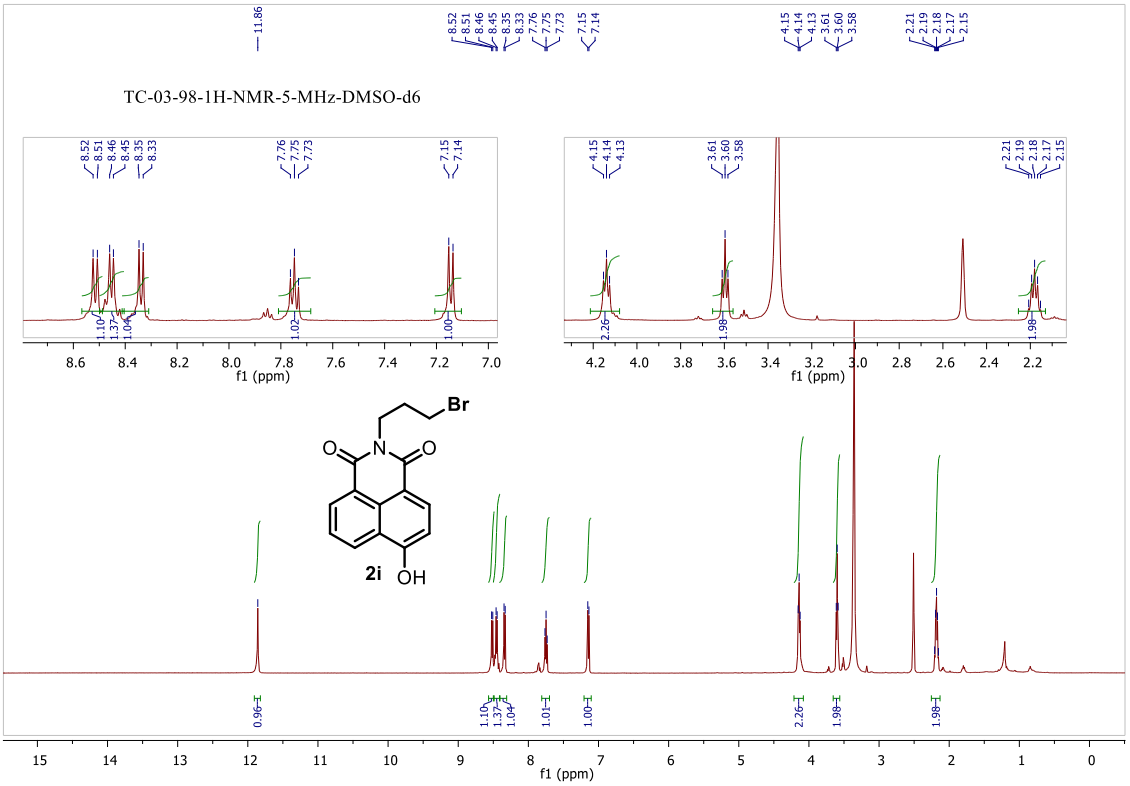
Acquisition Parameter

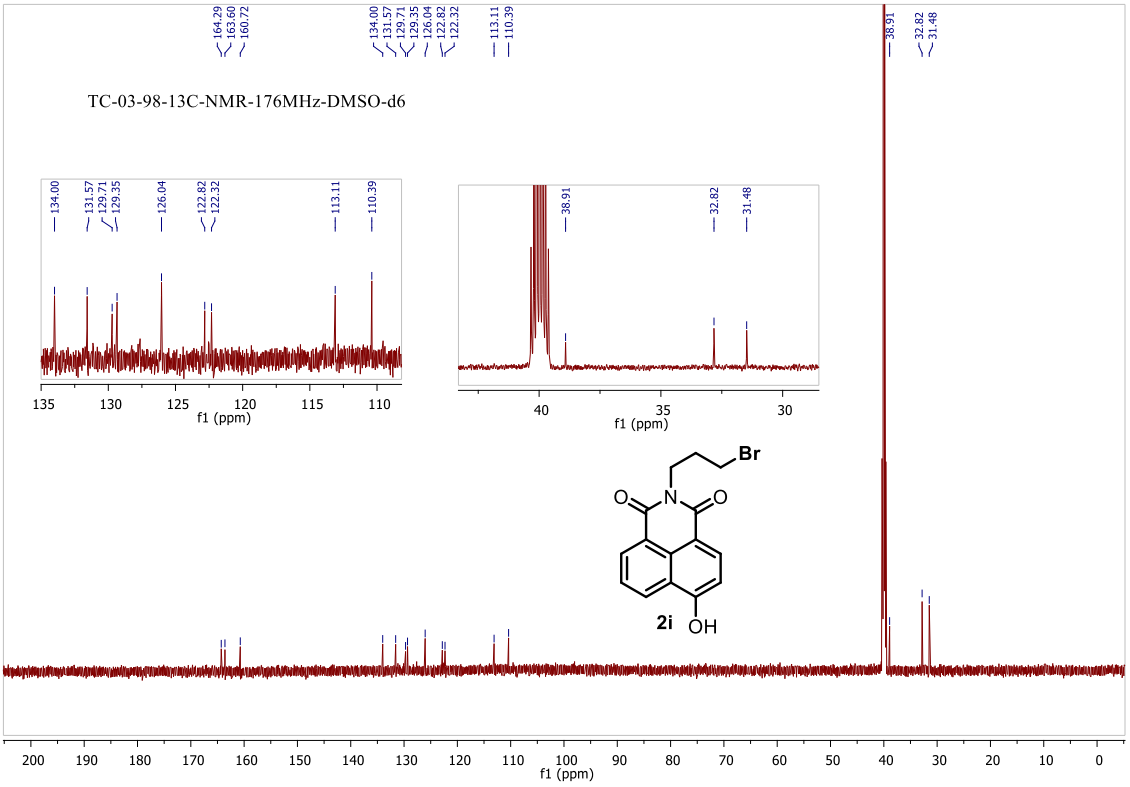
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Source








TC-03-98-1H-NMR-5-MHz-DMSO-d6
Br
O
N
O
2i
OH
f1 (ppm)


TC-03-98-13C-NMR-176MHz-DMSO-d6
Br
O
N
O
2i
OH
f1 (ppm)

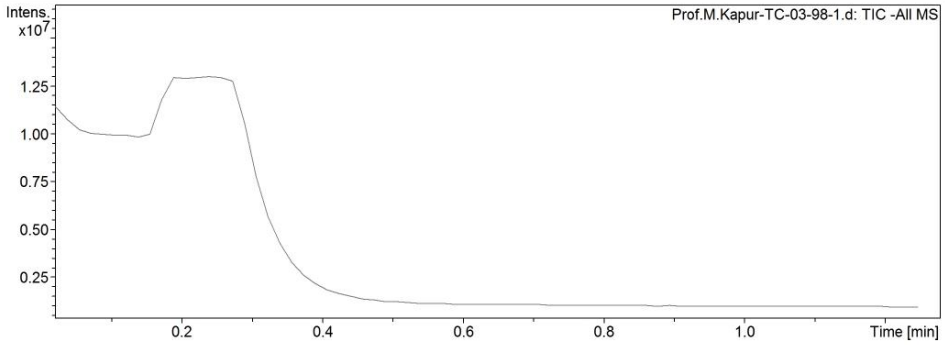
Display Report

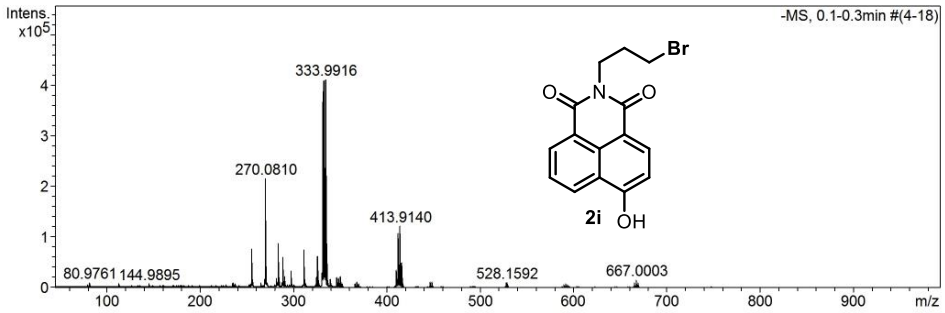
Analysis Info

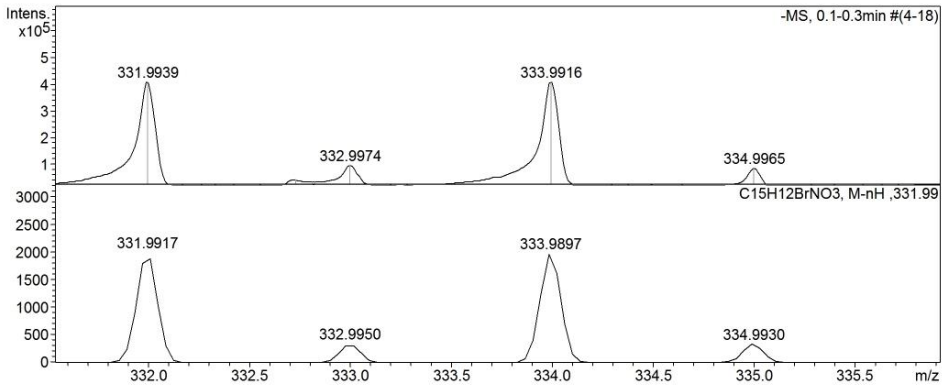
Analysis Name	D:\Data\USER DATA 2023\July\21-july\Prof.M.Kapur-TC-03-98-1.d	Acquisition Date	21-07-2023 12:24:28
Method	tune_low_neg_Jan23.m	Operator	Bruker
Sample Name	TC-03-98-1	Instrument	micrOTOF-Q 10330
Comment			

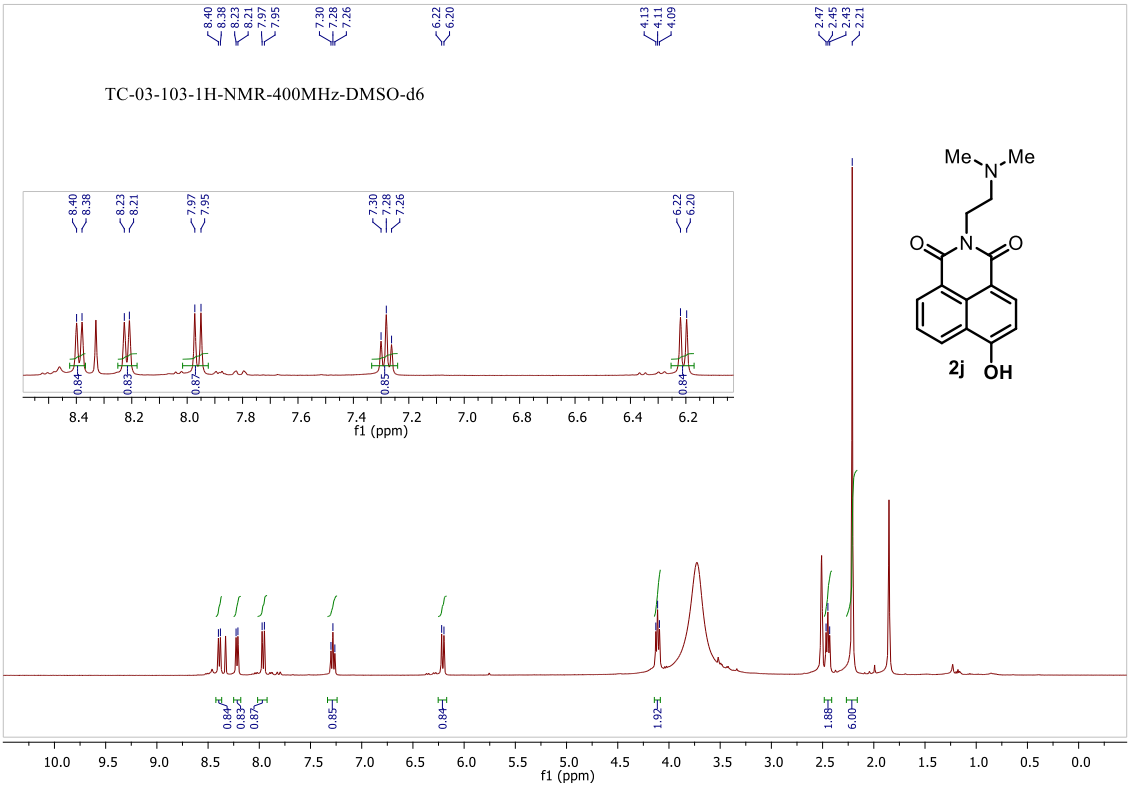
Acquisition Parameter

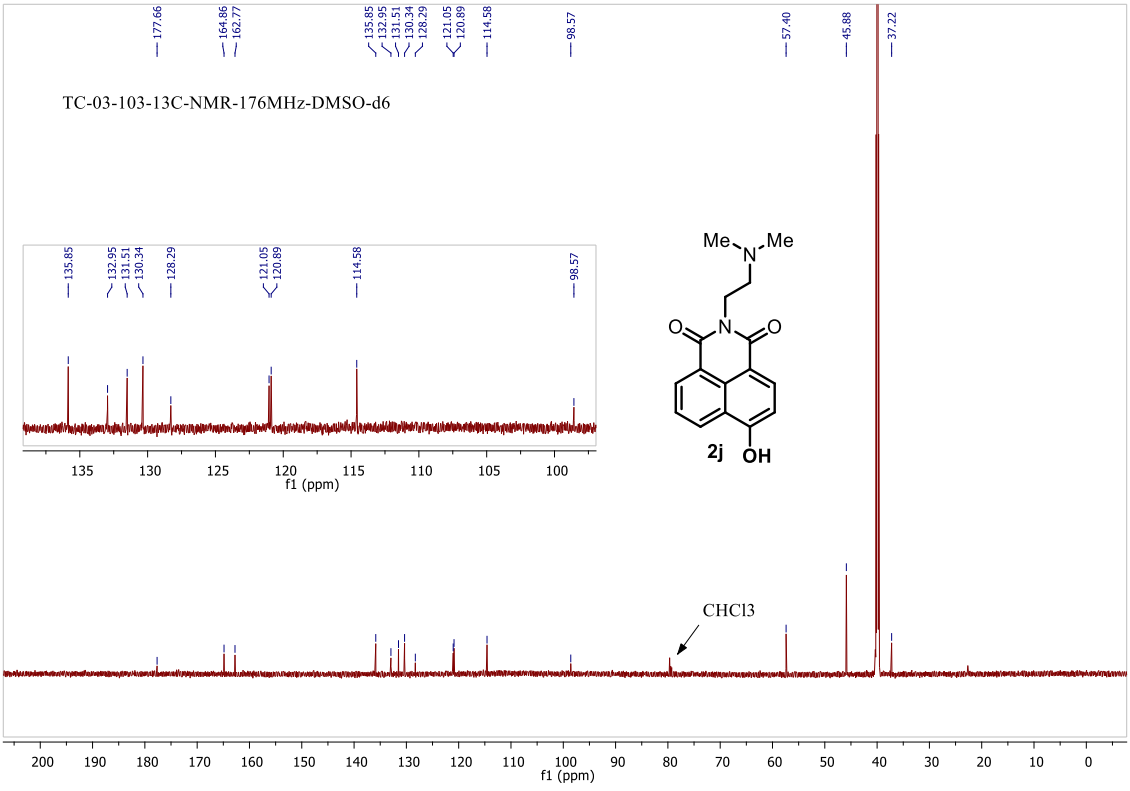
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.6 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste








TC-03-103-1H-NMR-400MHz-DMSO-d6
Me
N
Me
O
N
O
2j
OH
f1 (ppm)


TC-03-103-13C-NMR-176MHz-DMSO-d6
Me
N
Me
O
N
O
2j
OH
CHCl3
f1 (ppm)

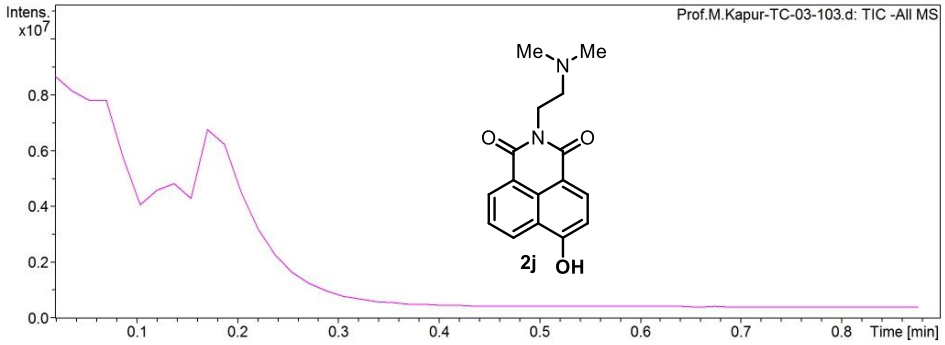
Display Report

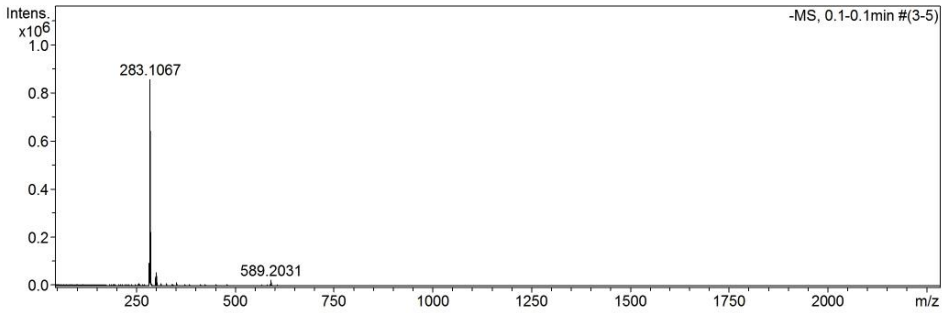
Analysis Info

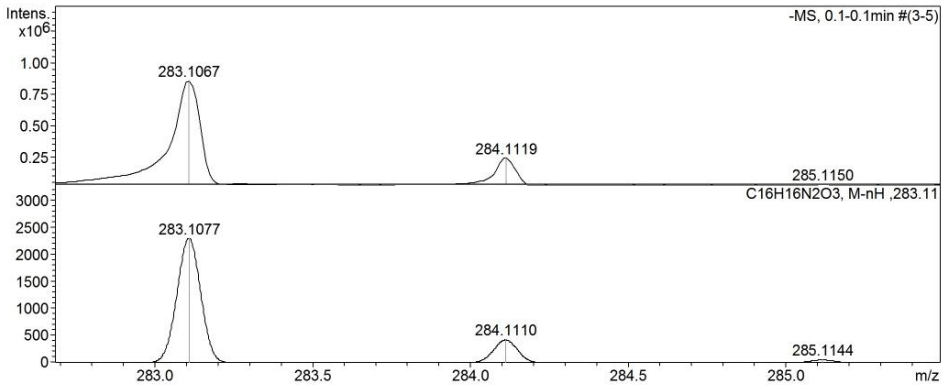
Analysis Name	D:\Data\USER DATA 2023\July\25-july\Prof.M.Kapur-TC-03-103.d	Acquisition Date	25-07-2023 15:46:44
Method	tune_low_neg.new.m	Operator	Bruker
Sample Name	TC-03-103	Instrument	micrOTOF-Q 10330
Comment			

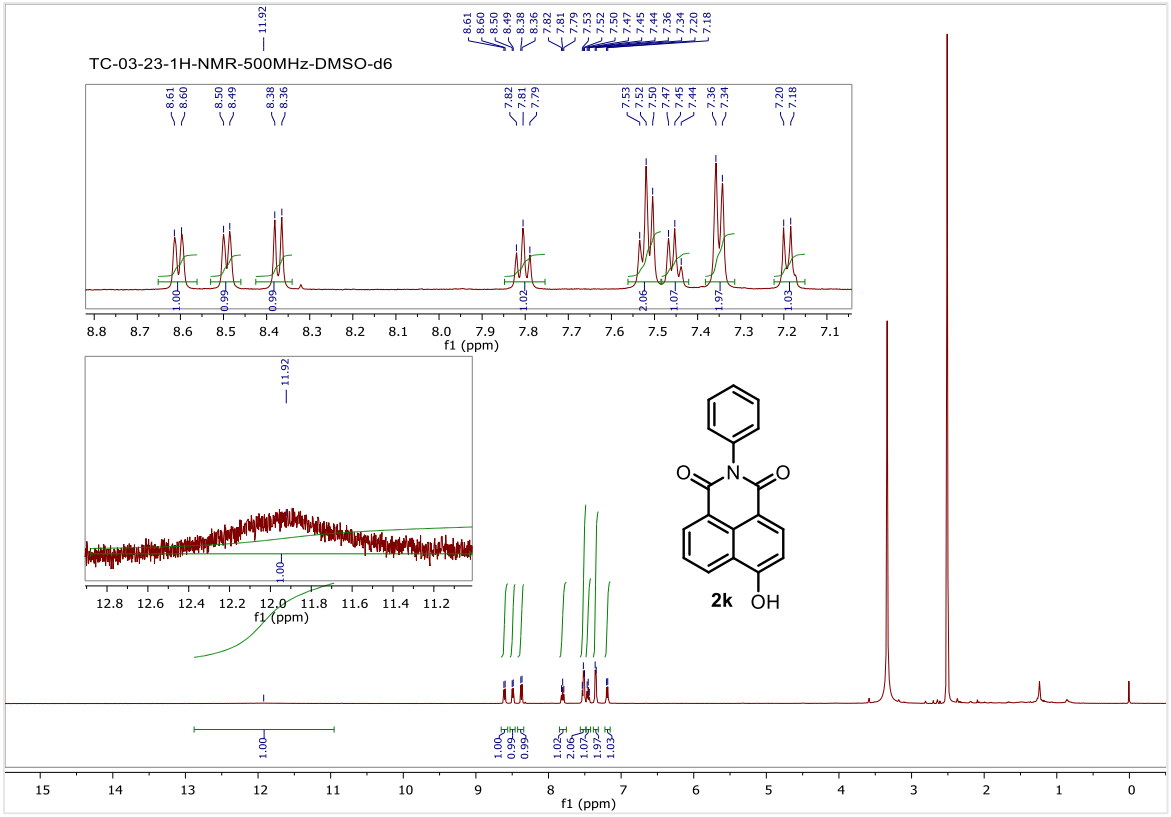
Acquisition Parameter

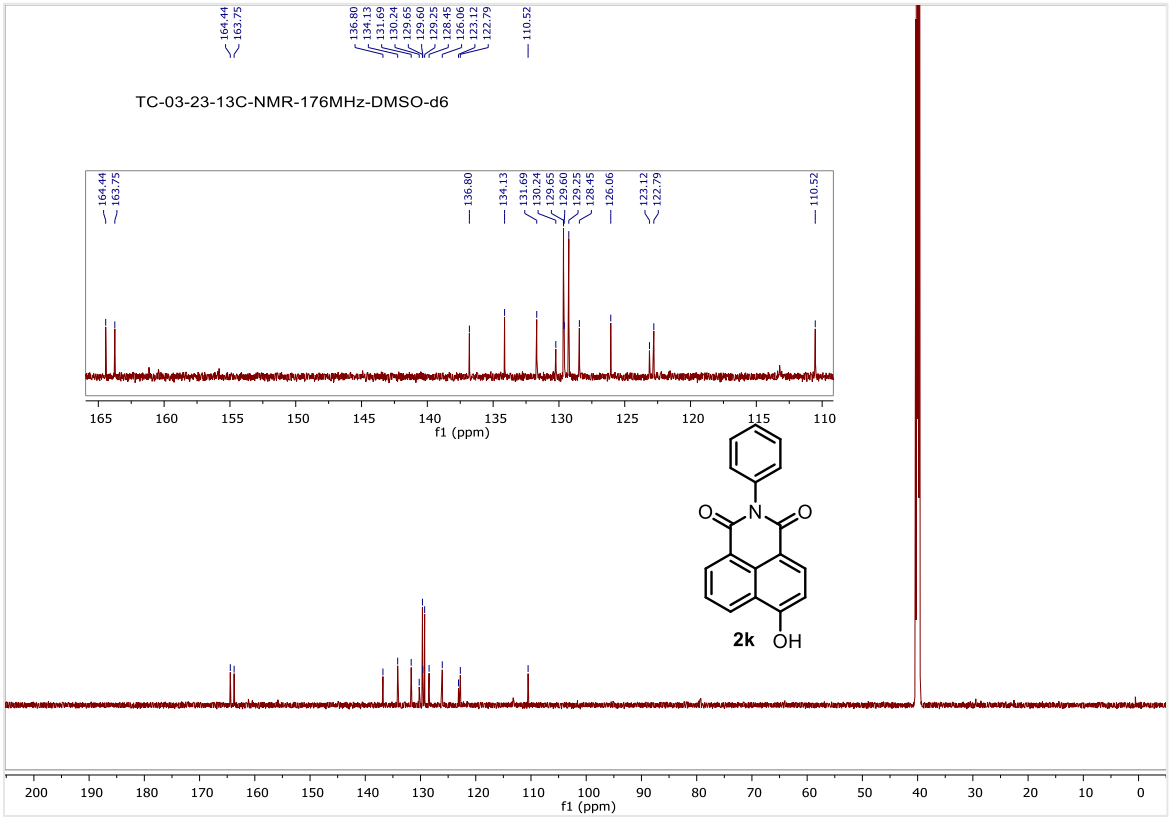
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste








TC-03-23-1H-NMR-500MHz-DMSO-d6
11.92
8.61
8.60
8.50
8.49
8.38
8.36
7.82
7.81
7.79
7.53
7.52
7.50
7.47
7.45
7.44
7.36
7.34
7.20
7.18
1.00
0.99
0.99
1.02
2.06
1.07
1.97
1.03
f1 (ppm)
O
N
O
2k
OH


TC-03-23-13C-NMR-176MHz-DMSO-d6
164.44
163.75
136.80
134.13
131.69
130.24
129.65
129.60
129.25
128.45
126.06
123.12
122.79
110.52
f1 (ppm)
O
N
O
2k
OH

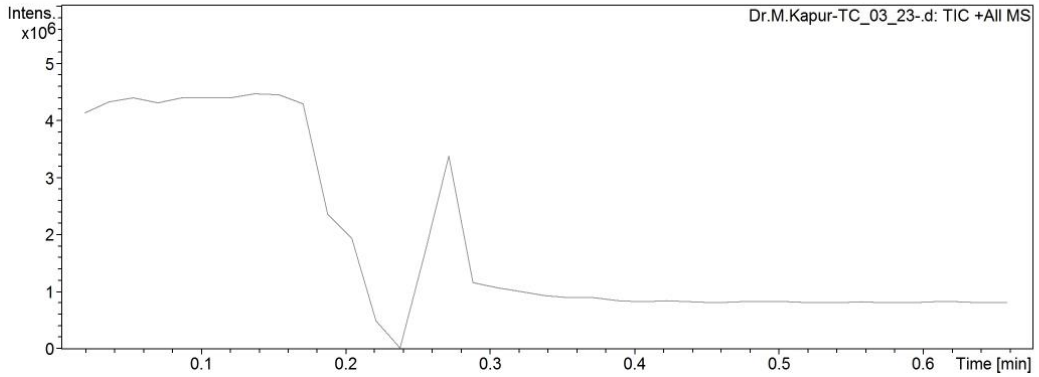
Display Report

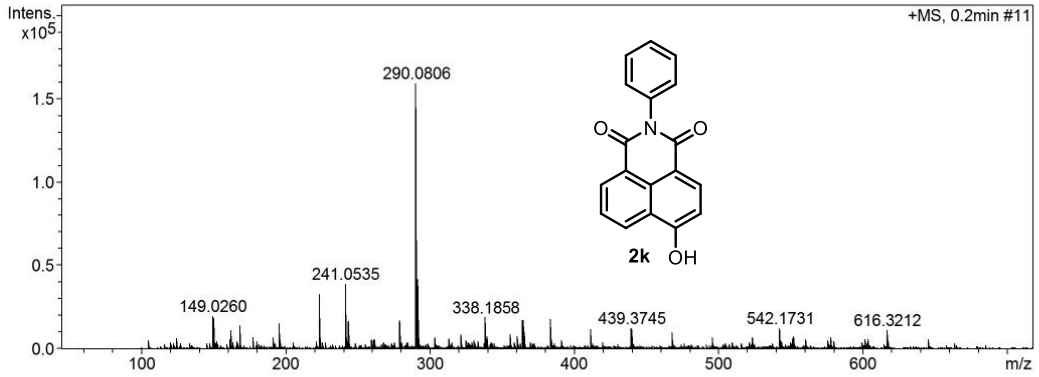
Analysis Info

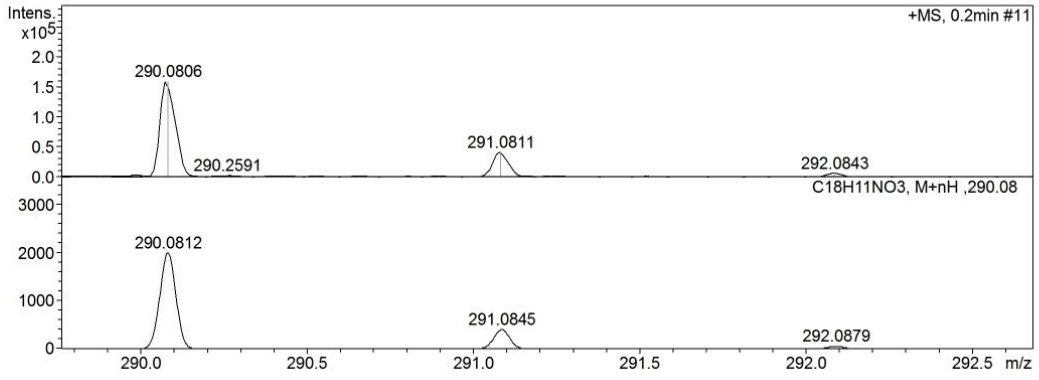
Analysis Name	D:\Data\USER DATA 2023\Jan\13-01-2023\Dr.M.Kapur-TC_03_23-.d	Acquisition Date	13-01-2023 15:51:36
Method	tune_low_Jan23.m	Operator	Bruker
Sample Name	TC_03_23-	Instrument	micrOTOF-Q 10330
Comment			

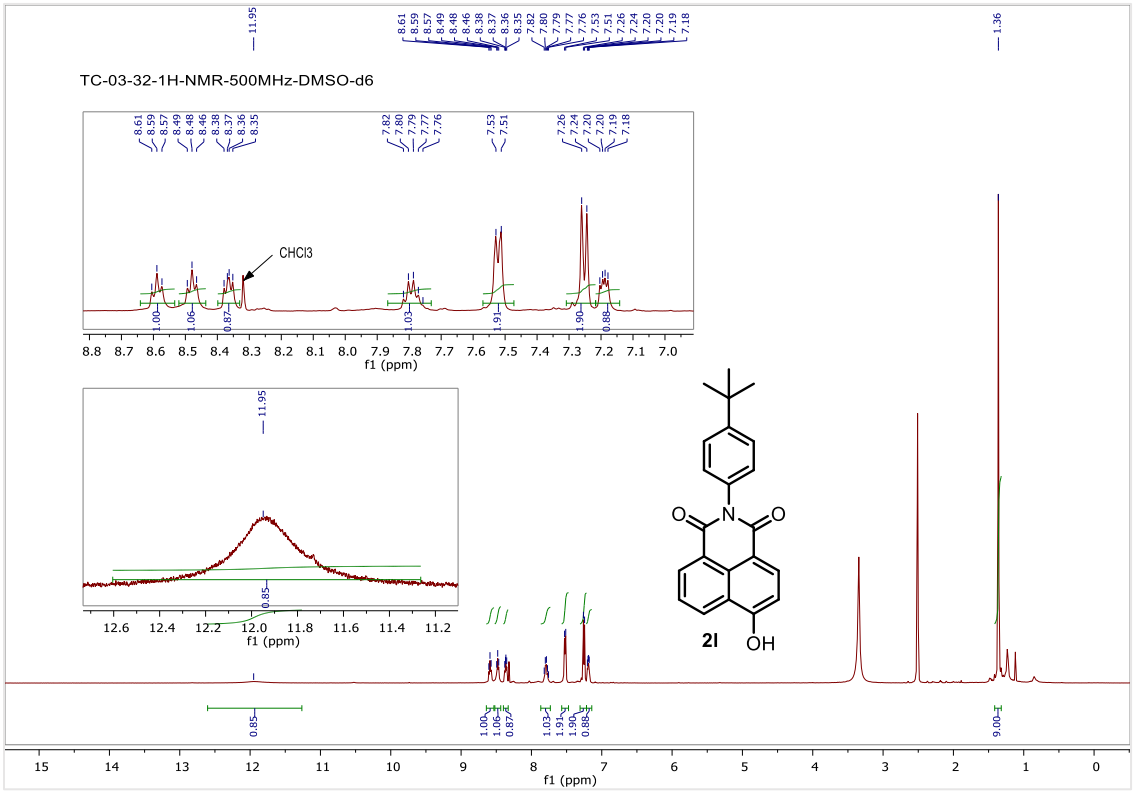
Acquisition Parameter

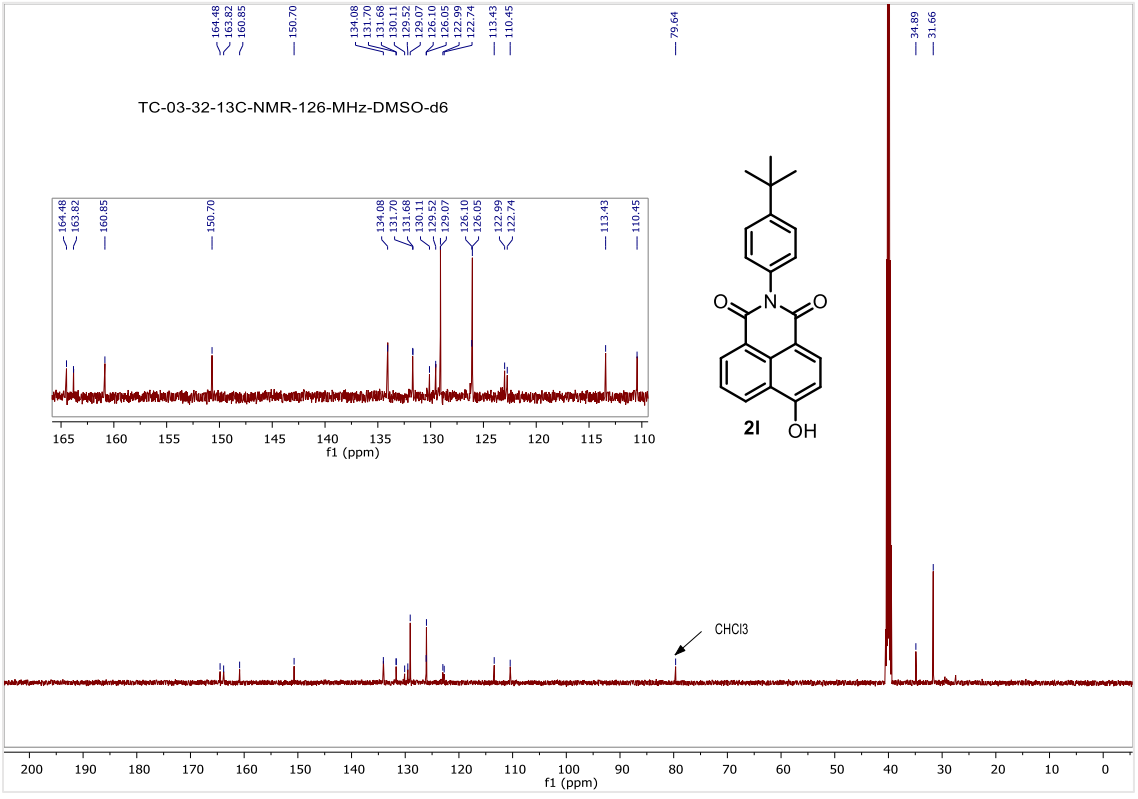
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste








TC-03-32-1H-NMR-500MHz-DMSO-d6
CHCl3
f1 (ppm)
2l
OH


TC-03-32-13C-NMR-126-MHz-DMSO-d6
CHCl3
f1 (ppm)
2l
OH

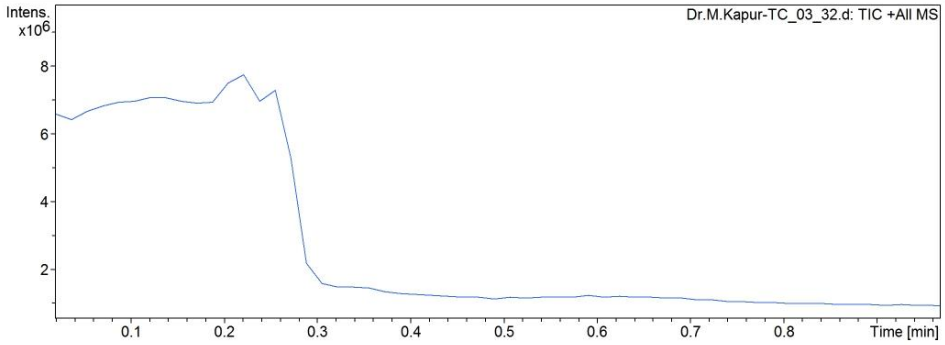
Display Report

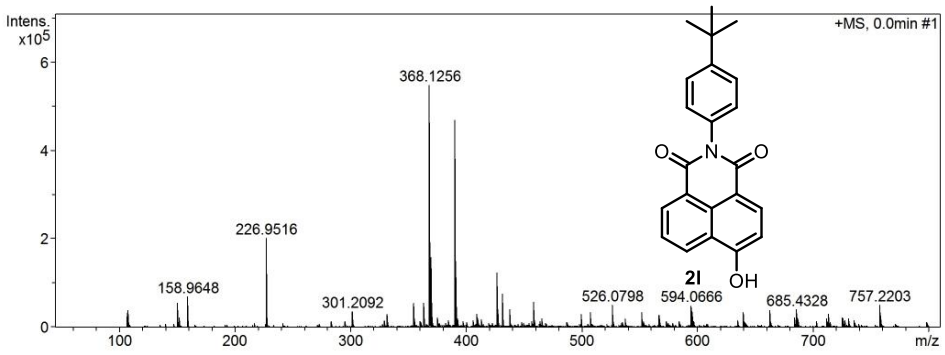
Analysis Info

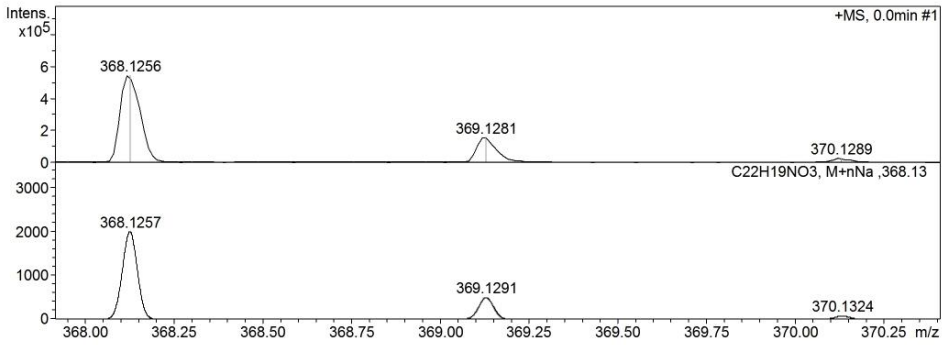
Analysis Name	D:\Data\USER DATA 2023\Jan\25-01-2023\Dr.M.Kapur-TC_03_32.d	Acquisition Date	25-01-2023 15:39:17
Method	tune_low_Jan23.m	Operator	Bruker
Sample Name	TC_03_32	Instrument	micrOTOF-Q 10330
Comment			

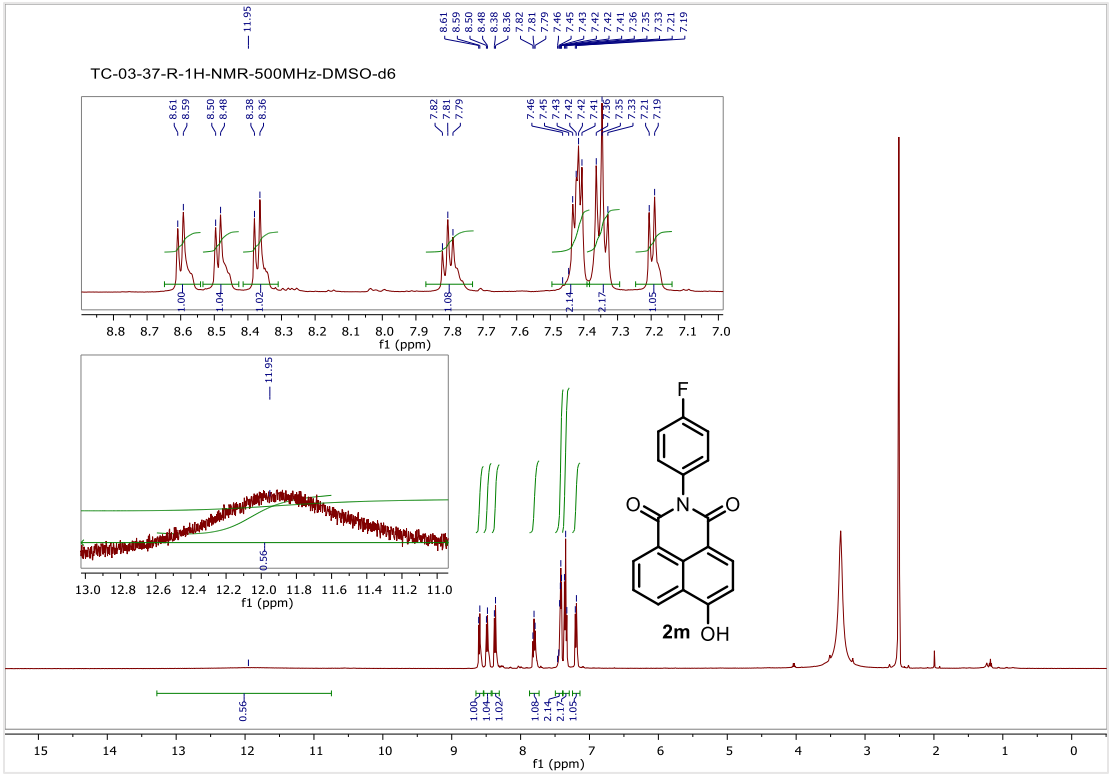
Acquisition Parameter

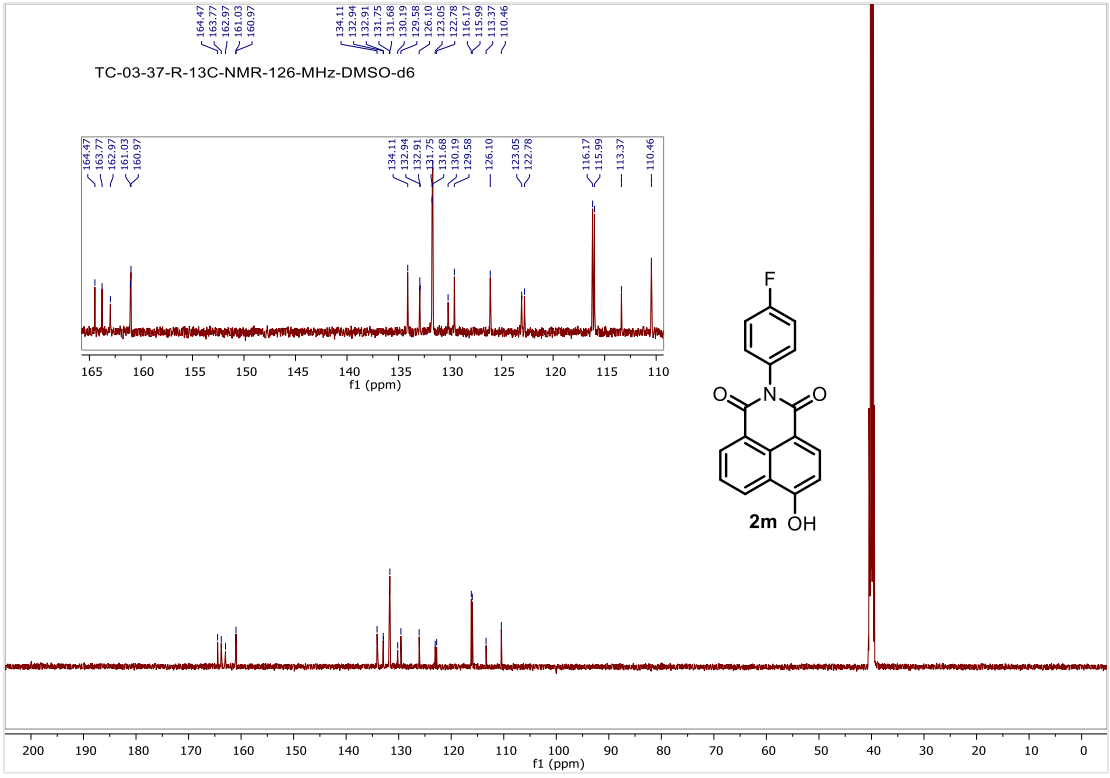
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

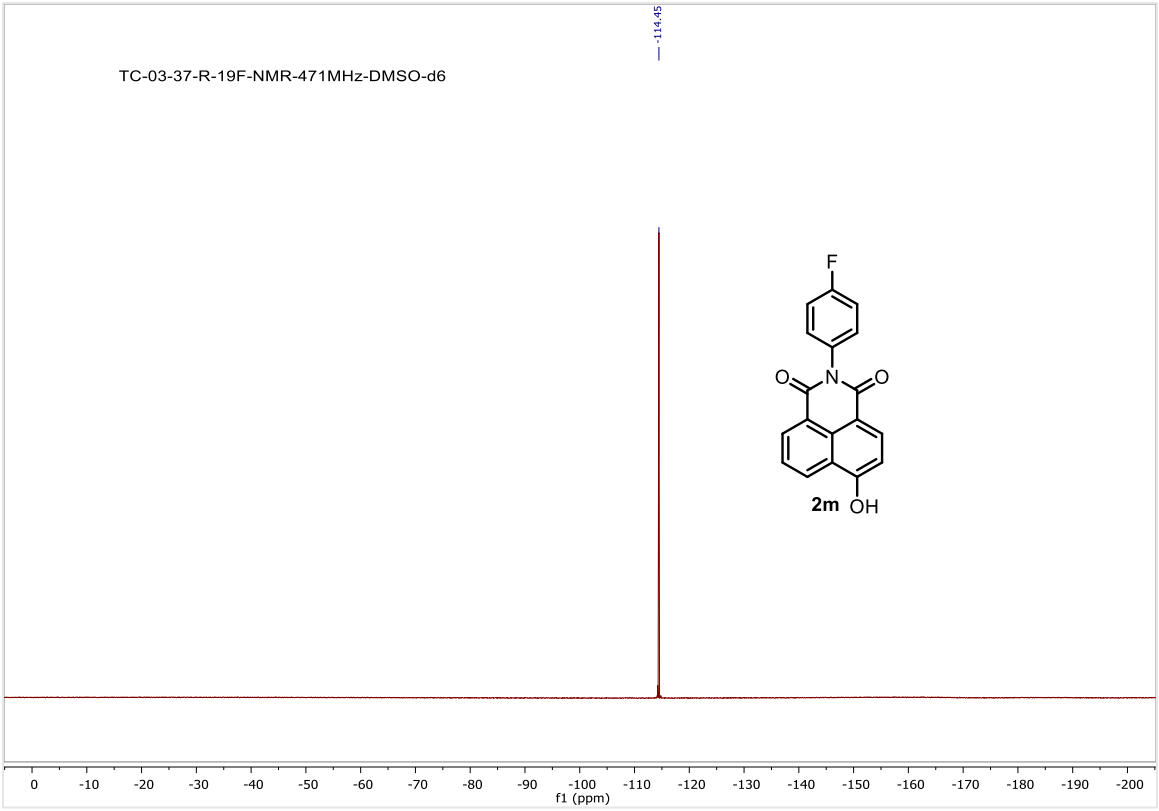







TC-03-37-R-1H-NMR-500MHz-DMSO-d6
f1 (ppm)
F
O
N
O
2m
OH


TC-03-37-R-13C-NMR-126-MHz-DMSO-d6
f1 (ppm)
F
O
N
O
2m
OH


TC-03-37-R-19F-NMR-471MHz-DMSO-d6
-114.45
F
O
N
O
2m
OH
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

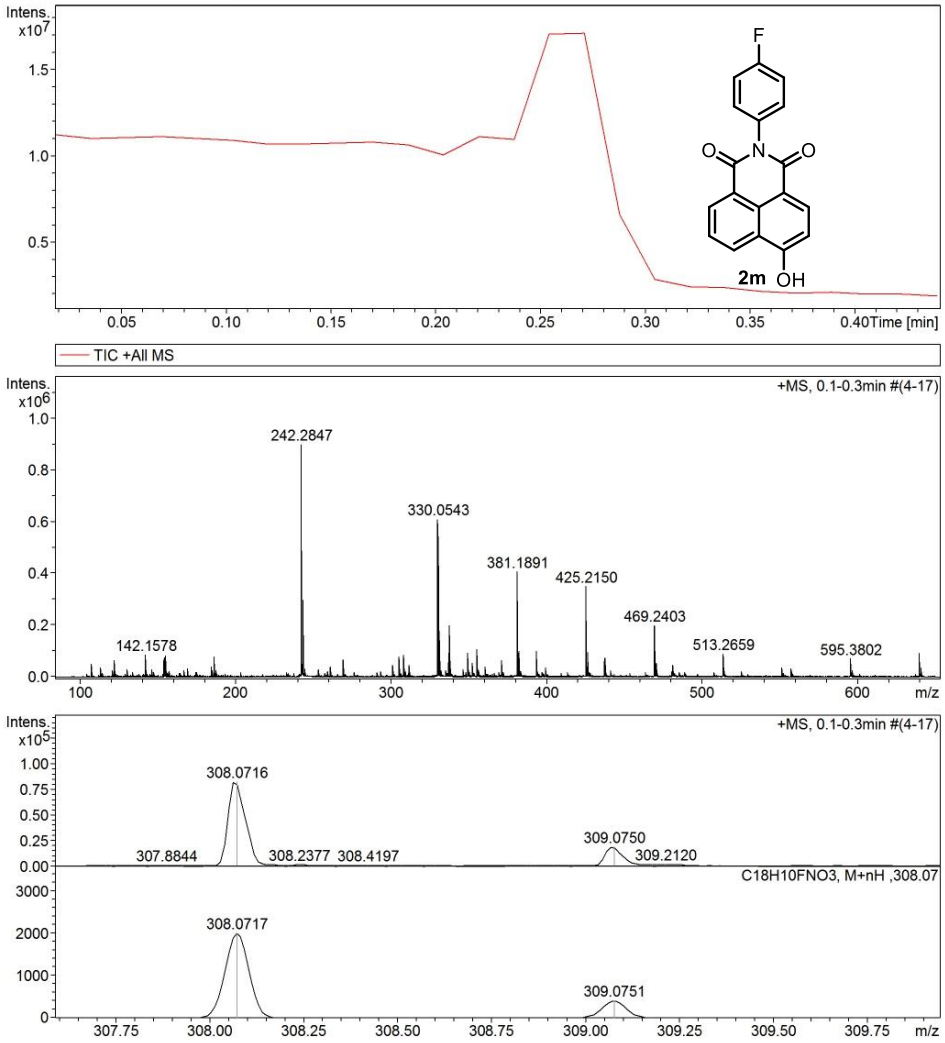
Display Report

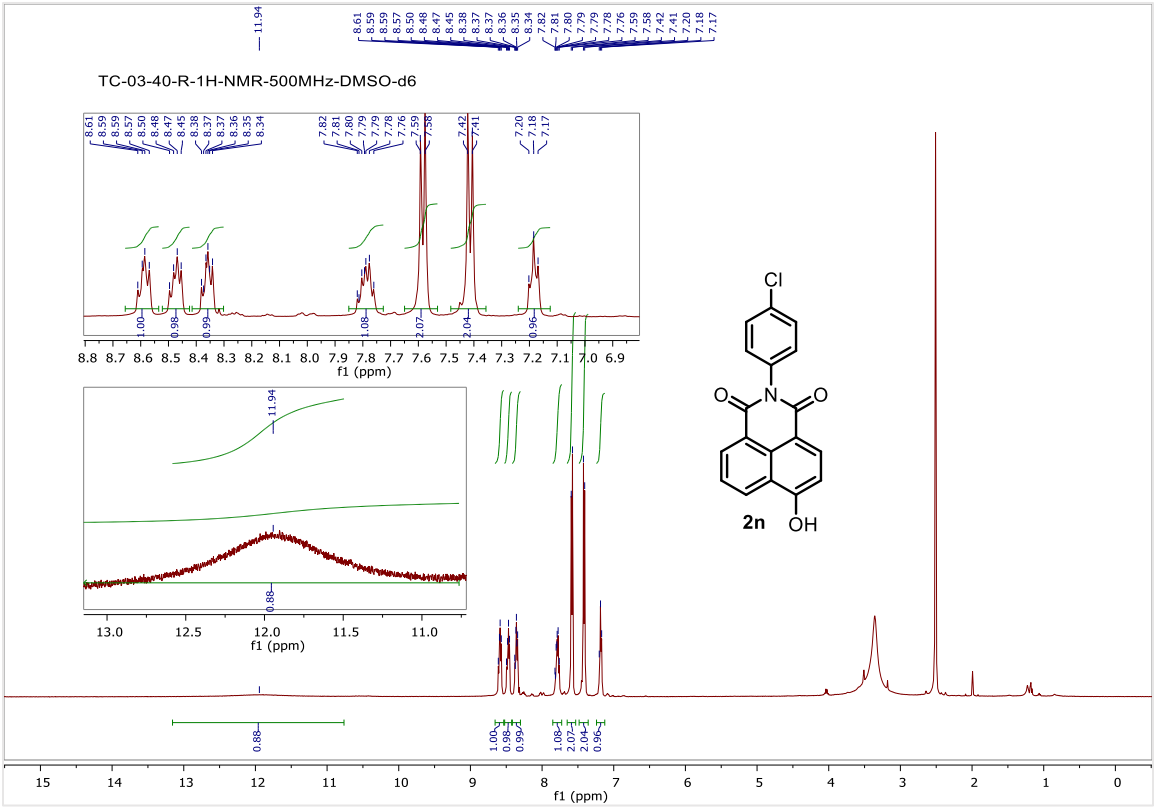
Analysis Info

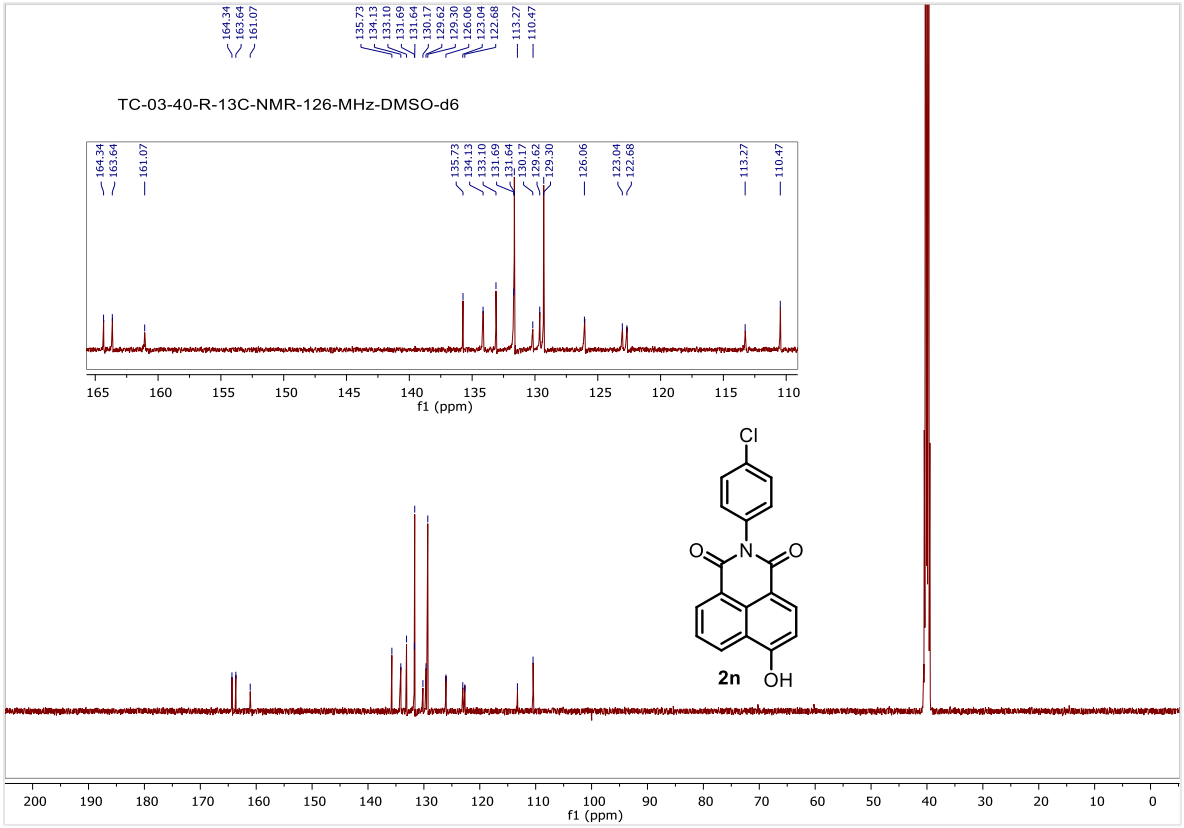
Analysis Name	D:\Data\USER DATA 2023\Feb\20-02-2023\Prof M Kapur-TC-03-37-.d	Acquisition Date	20-02-2023 10:48:34
Method	tune_low_Jan23.m	Operator	Bruker
Sample Name	TC-03-37-	Instrument	micrOTOF-Q 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.8 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.5 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste




TC-03-40-R-1H-NMR-500MHz-DMSO-d6
f1 (ppm)
Cl
O
N
O
2n
OH


TC-03-40-R-13C-NMR-126-MHz-DMSO-d6
f1 (ppm)
Cl
O
N
O
2n
OH

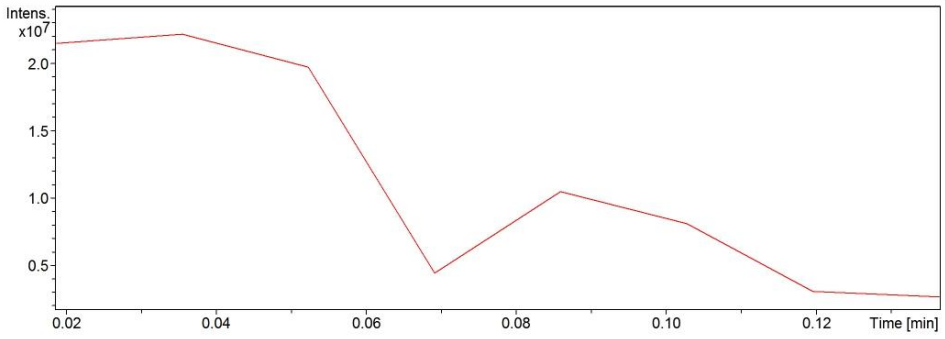
Display Report

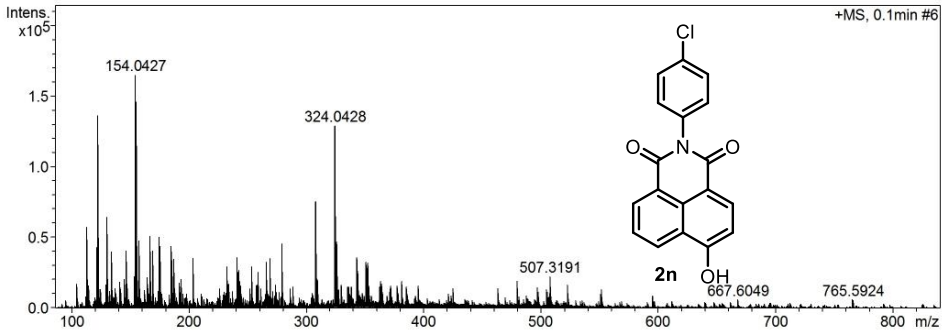
Analysis Info

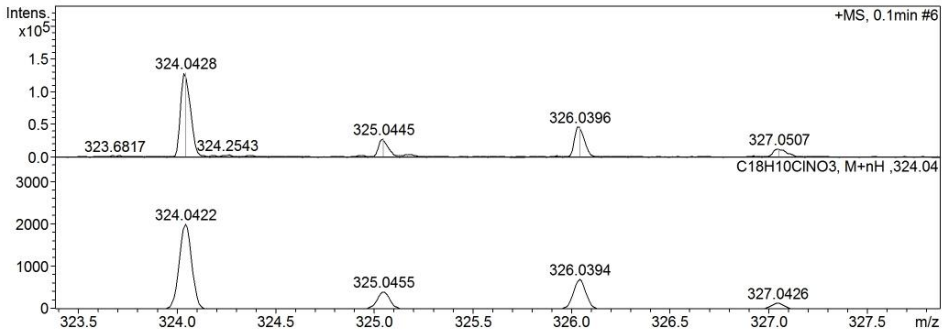
Analysis Name	D:\Data\USER DATA 2023\Feb\20-02-2023\Prof M Kapur-TC-03-40.d	Acquisition Date	20-02-2023 11:00:11
Method	tune_low_Jan23.m	Operator	Bruker
Sample Name	TC-03-40	Instrument	micrOTOF-Q 10330
Comment			

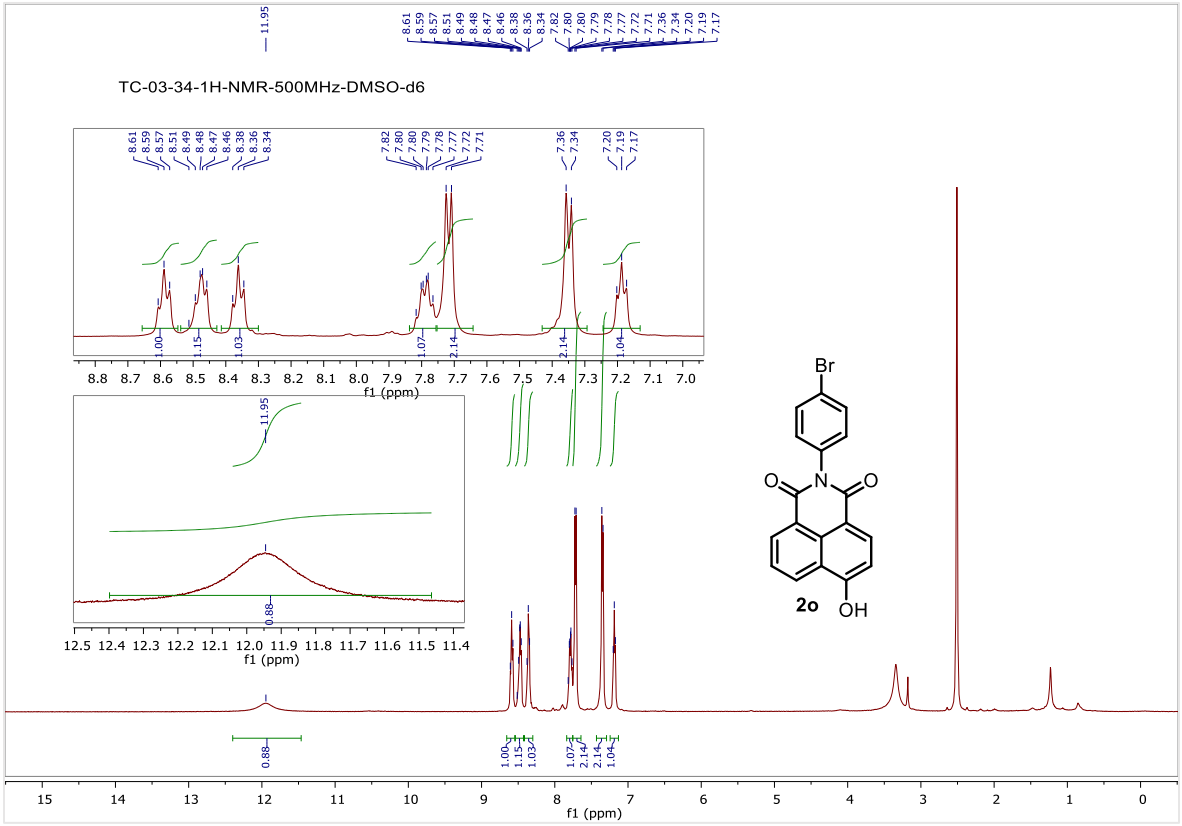
Acquisition Parameter

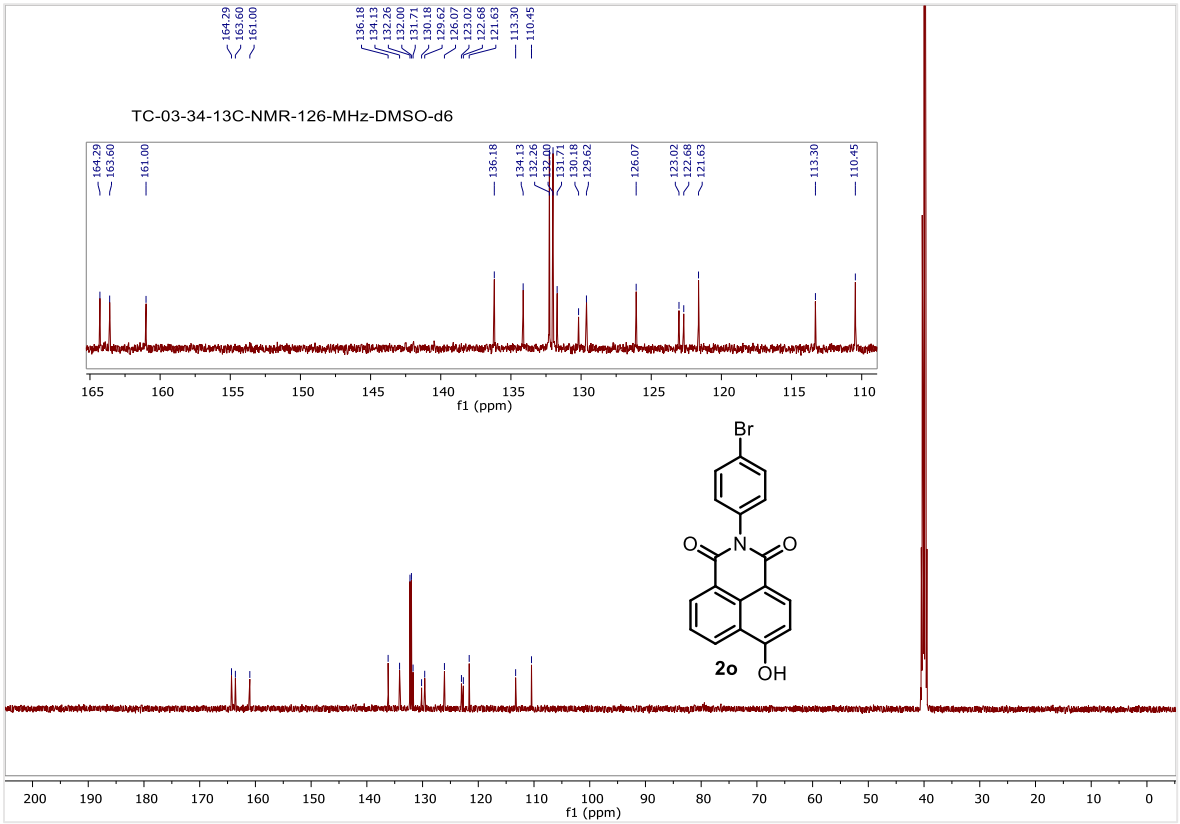
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.8 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.5 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste








TC-03-34-1H-NMR-500MHz-DMSO-d6
Br
O
N
O
2o
OH
f1 (ppm)


TC-03-34-13C-NMR-126-MHz-DMSO-d6
Br
O
N
O
2o
OH
f1 (ppm)

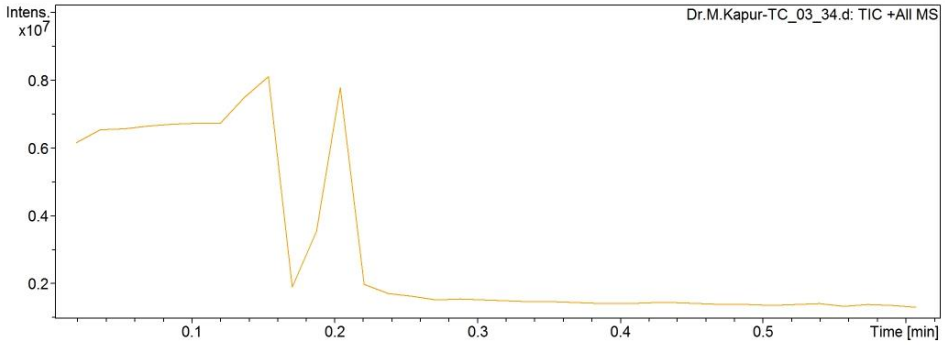
Display Report

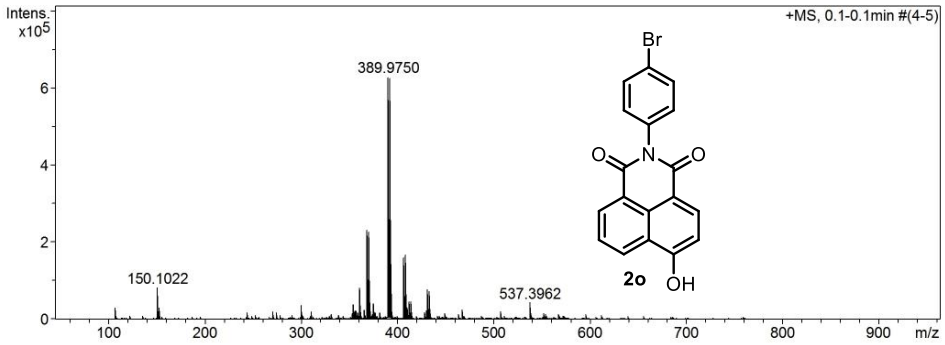
Analysis Info

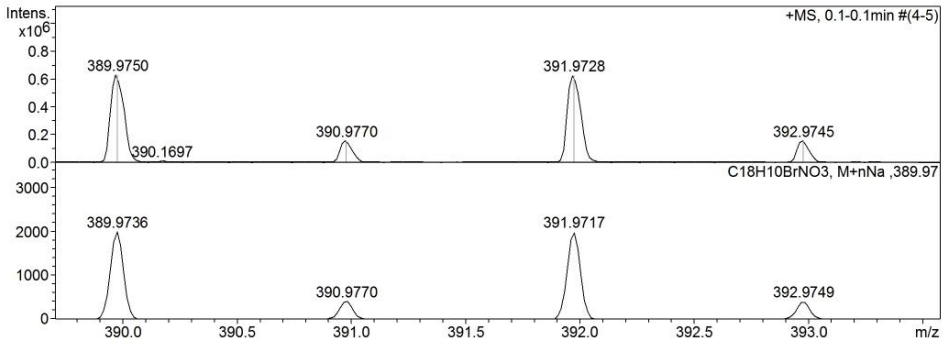
Analysis Name	D:\Data\USER DATA 2023\Jan\18-01-2023\Dr.M.Kapur-TC_03_34.d	Acquisition Date	18-01-2023 10:54:13
Method	tune_low_Jan23.m	Operator	Bruker
Sample Name	TC_03_34	Instrument	micrOTOF-Q 10330
Comment			

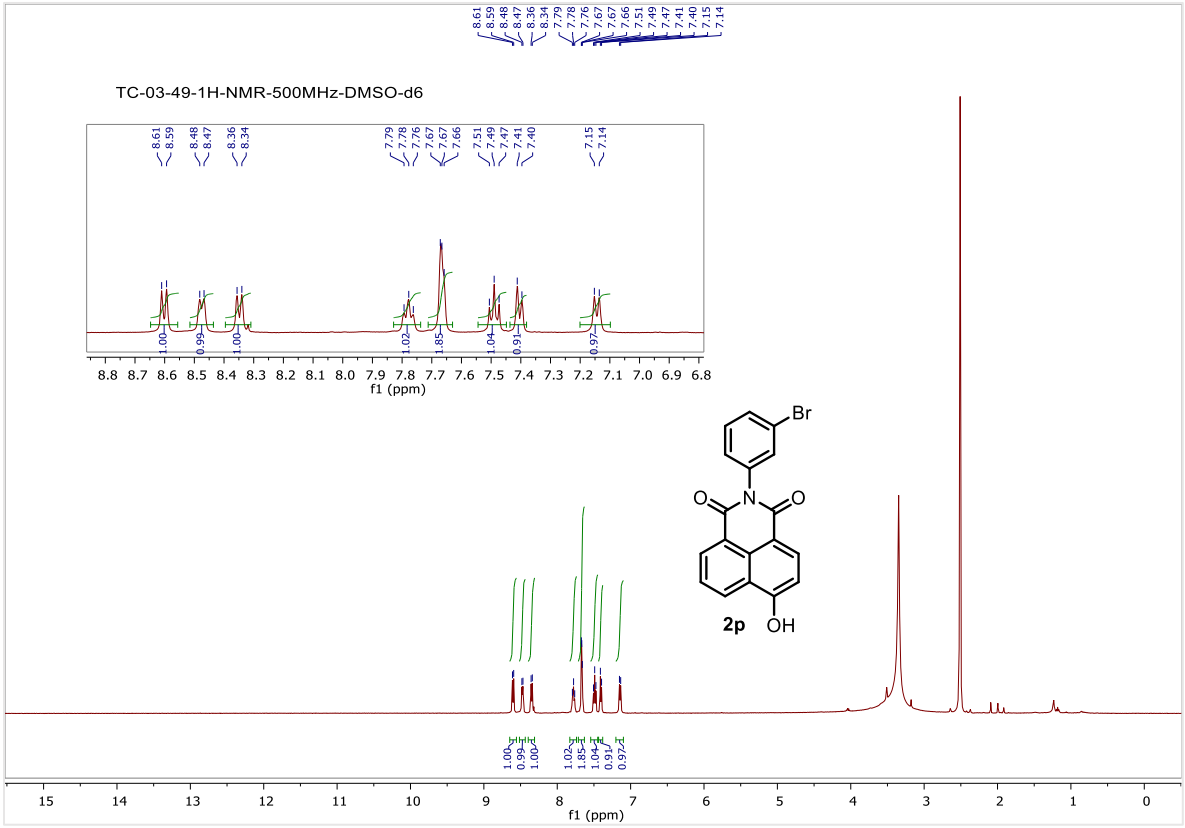
Acquisition Parameter

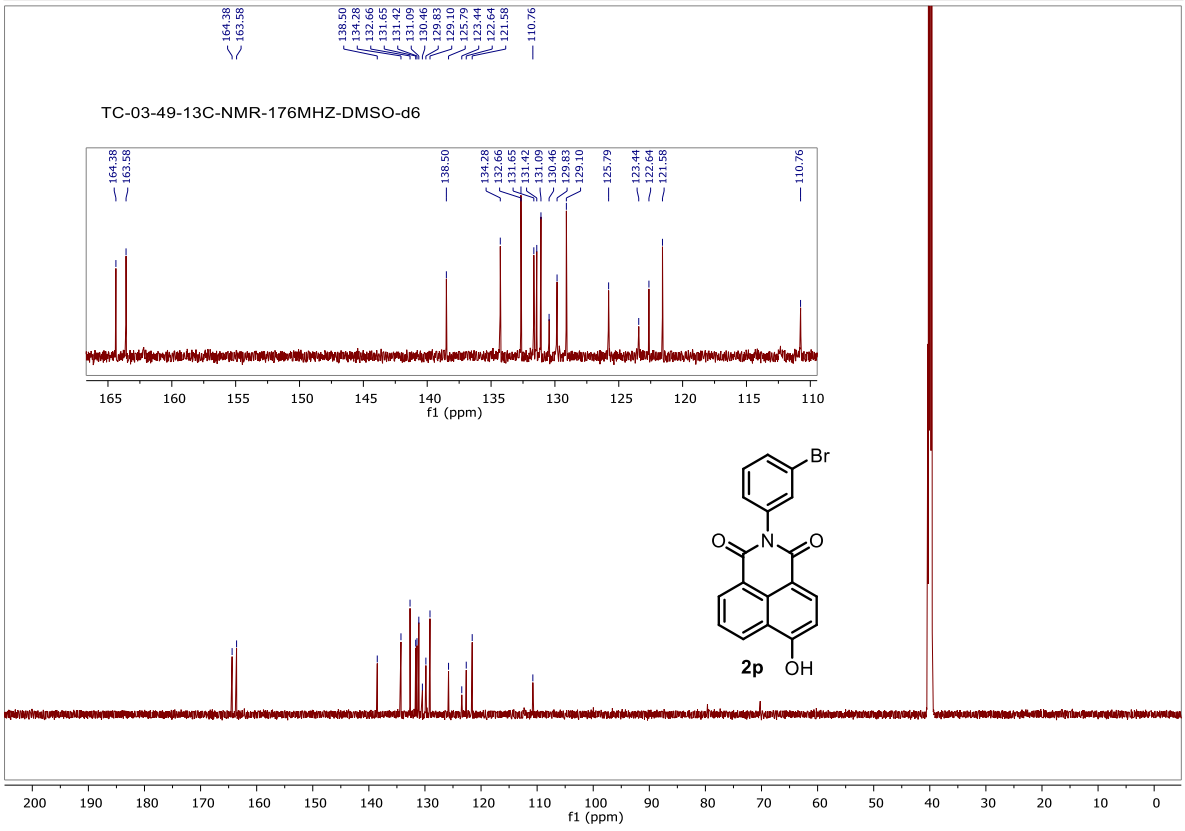
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste








TC-03-49-1H-NMR-500MHz-DMSO-d6
Br
O
N
O
2p
OH
f1 (ppm)


TC-03-49-13C-NMR-176MHZ-DMSO-d6
Br
O
N
O
2p
OH
f1 (ppm)

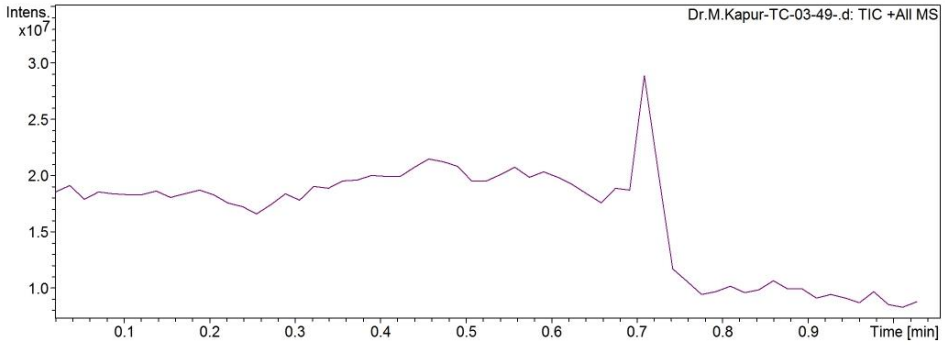
Display Report

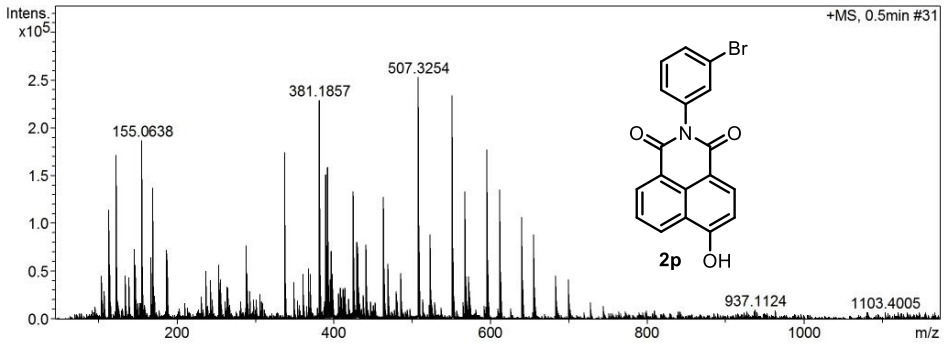
Analysis Info

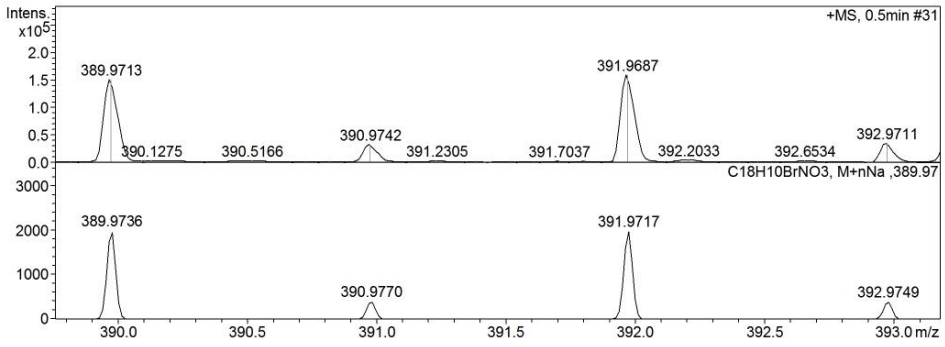
Analysis Name	D:\Data\USER DATA 2023\Feb\15-02-2023\Dr.M.Kapur-TC-03-49-.d	Acquisition Date	15-02-2023 14:17:15
Method	tune_low_Jan23.m	Operator	Bruker
Sample Name	TC-03-49-	Instrument	micrOTOF-Q 10330
Comment			

Acquisition Parameter

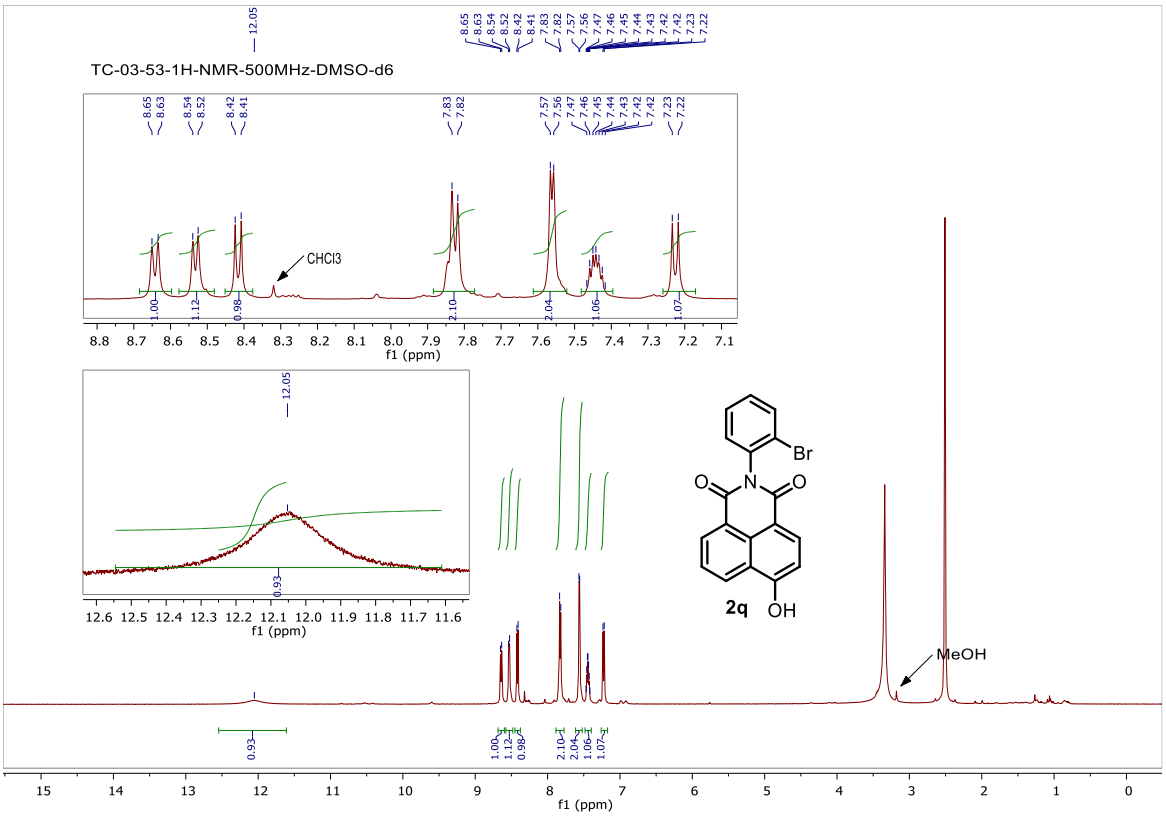
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

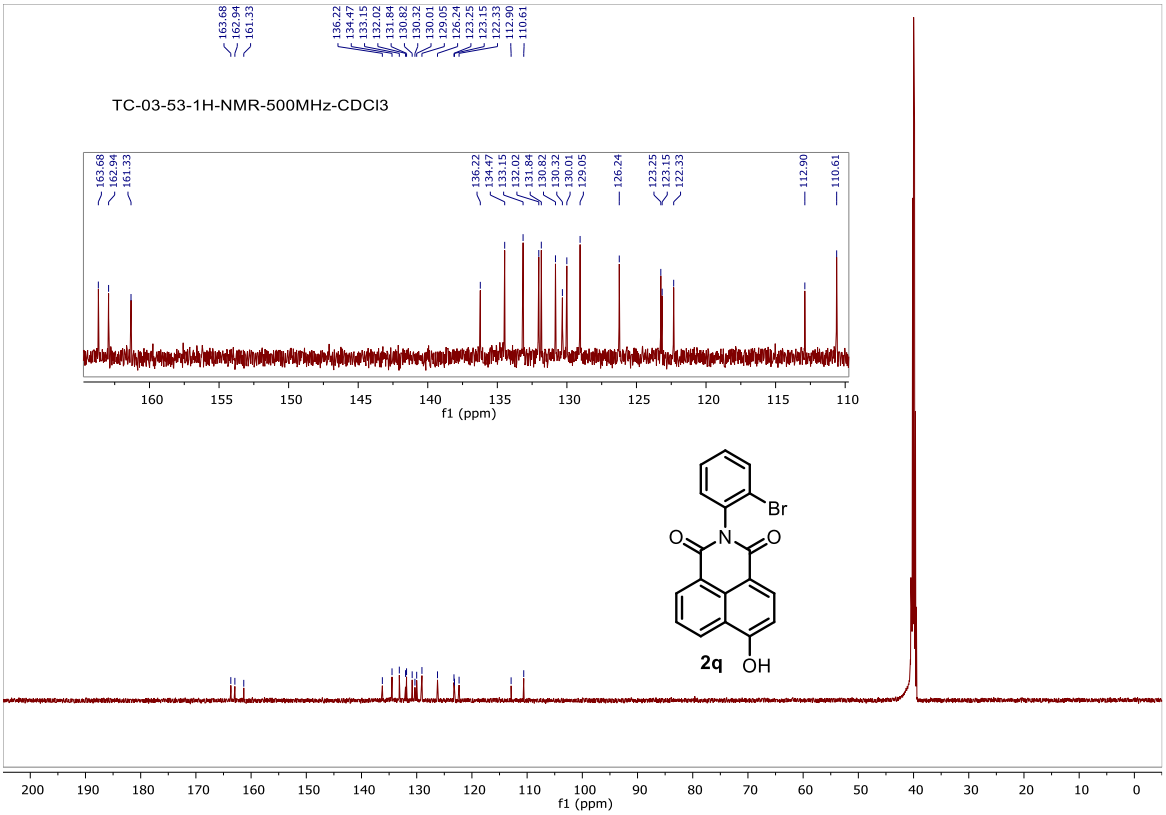






Bruker Compass DataAnalysis 4.0 printed: 15-02-2023 14:21:48 Page 1 of 1


TC-03-53-1H-NMR-500MHz-DMSO-d6
CHCl3
MeOH
Br
O
N
O
OH
2q
f1 (ppm)


TC-03-53-1H-NMR-500MHz-CDCl3
Br
O
N
O
OH
2q
f1 (ppm)

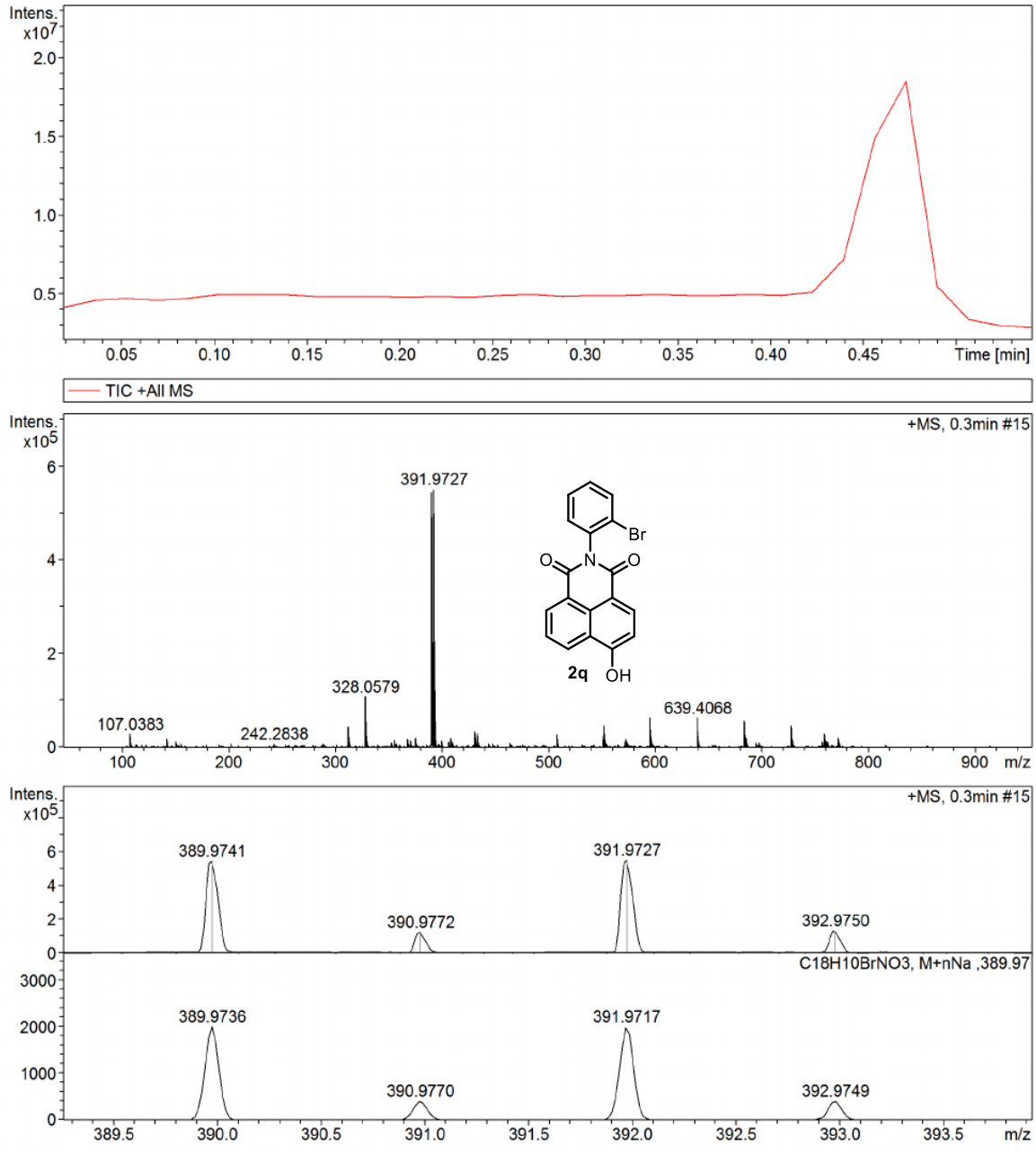
Display Report

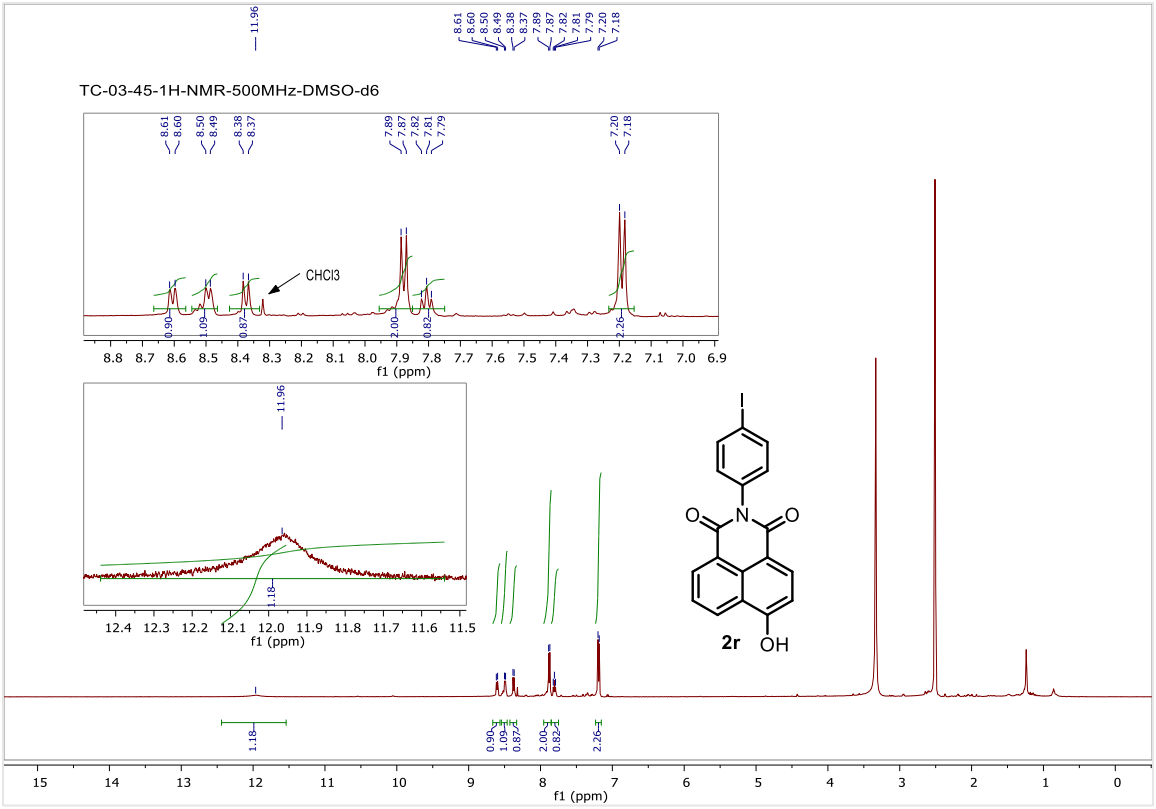
Analysis Info

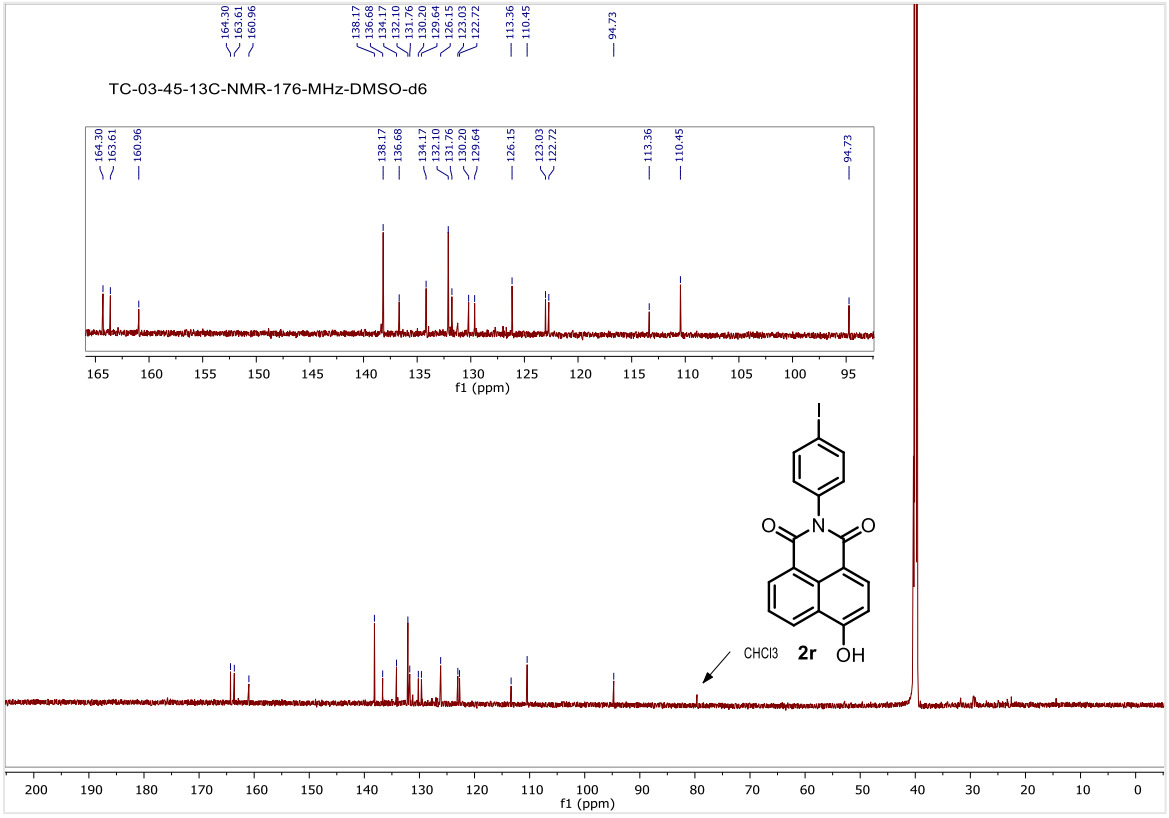
Analysis Name	D:\Data\USER DATA 2023\Feb\22-02-2023\Prof M Kapur-TC-03-53.d	Acquisition Date	22-02-2023 15:06:44
Method	tune_low_Jan23.m	Operator	Bruker
Sample Name	TC-03-53	Instrument	micrOTOF-Q 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.8 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste




TC-03-45-1H-NMR-500MHz-DMSO-d6
CHCl3
f1 (ppm)
2r
OH


TC-03-45-13C-NMR-176-MHz-DMSO-d6
f1 (ppm)
CHCl3
2r
OH

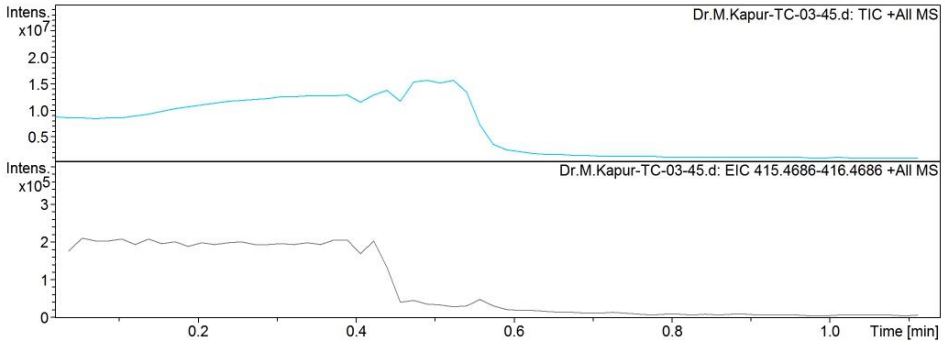
Display Report

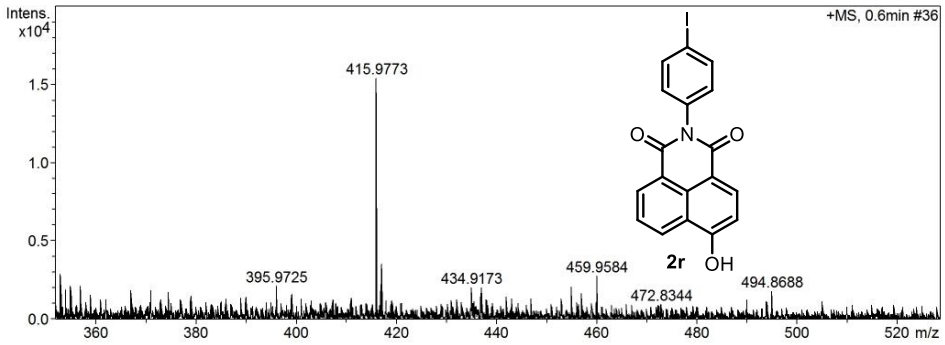
Analysis Info

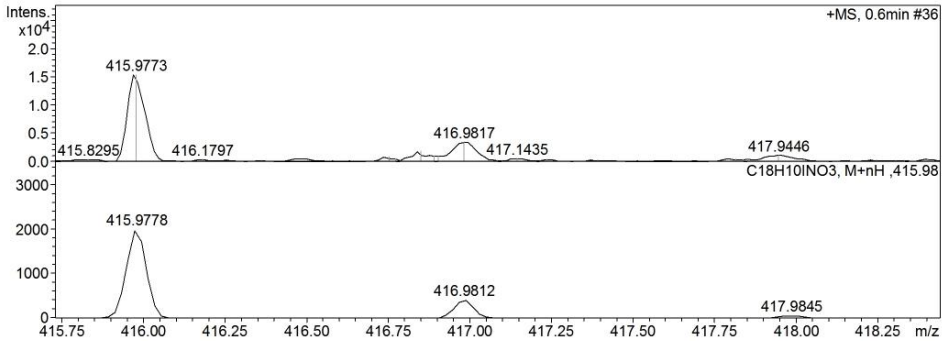
Analysis Name	D:\Data\USER DATA 2023\Feb\14-02-2023\Dr.M.Kapur-TC-03-45.d	Acquisition Date	14-02-2023 10:16:00
Method	tune_low_Jan23.m	Operator	Bruker
Sample Name	TC-03-45	Instrument	micrOTOF-Q 10330
Comment			

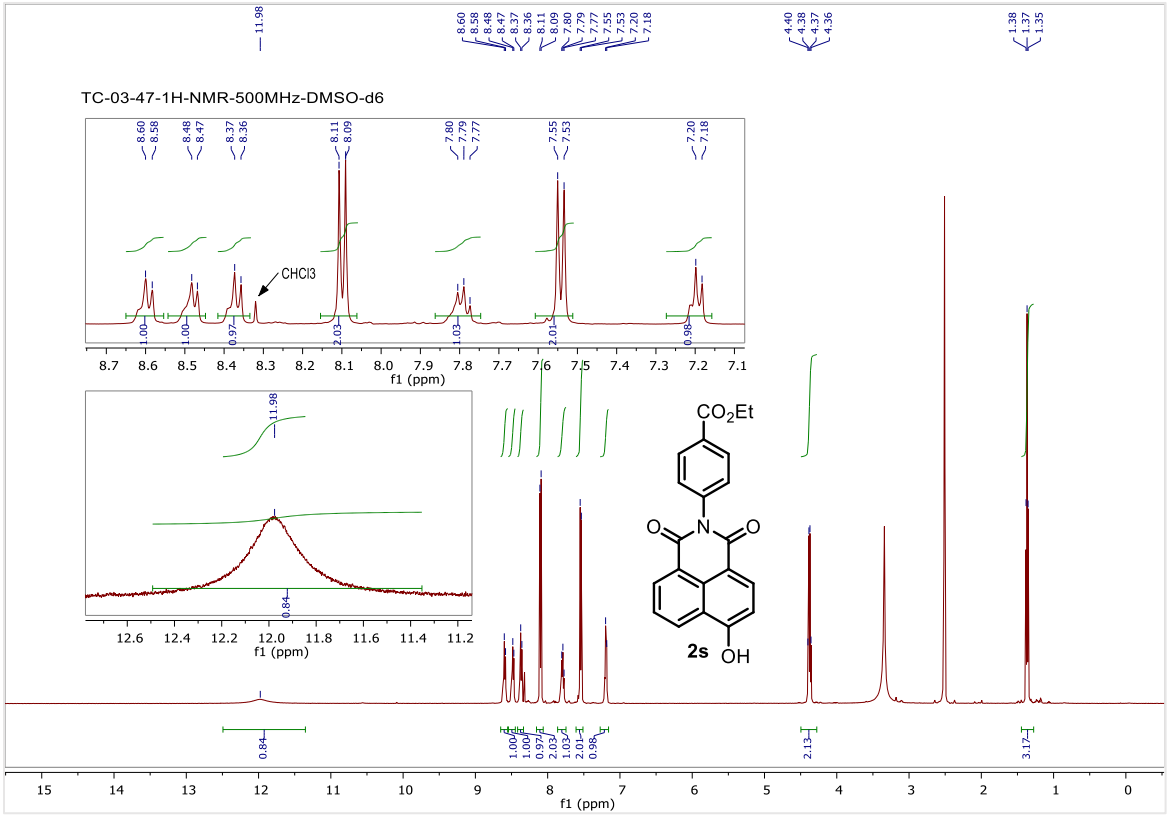
Acquisition Parameter

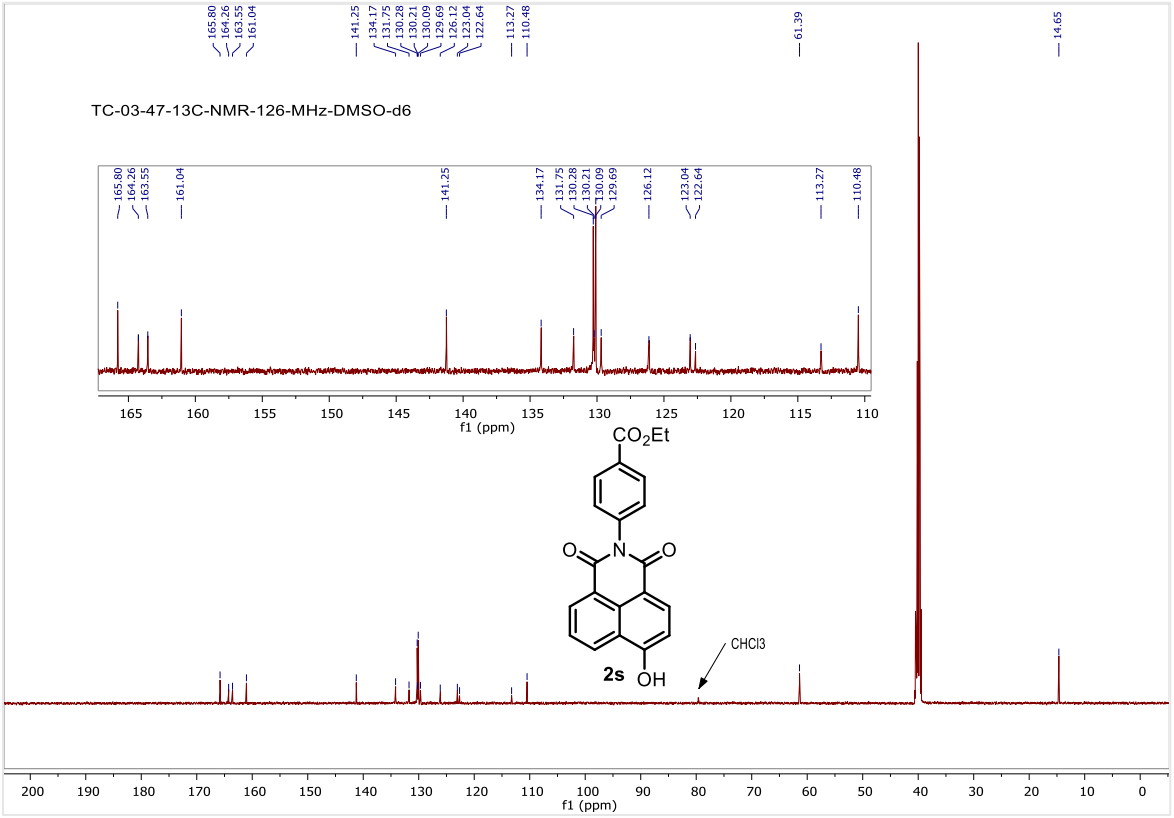
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste








TC-03-47-1H-NMR-500MHz-DMSO-d6
CHCl3
CO2Et
2s
OH
f1 (ppm)


TC-03-47-13C-NMR-126-MHz-DMSO-d6
CO2Et
2s
OH
CHCl3
f1 (ppm)

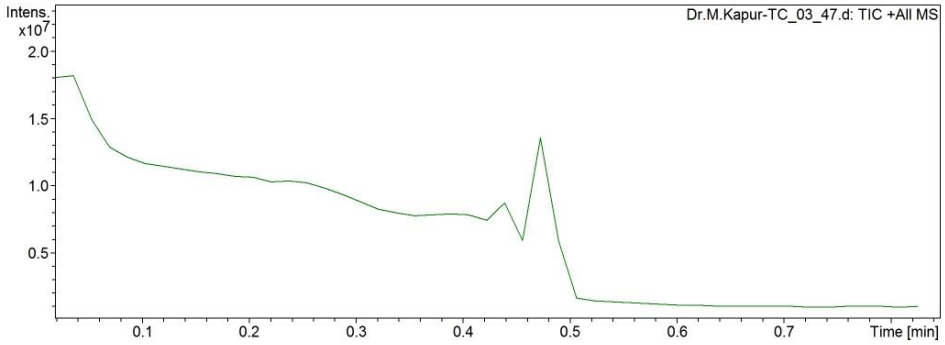
Display Report

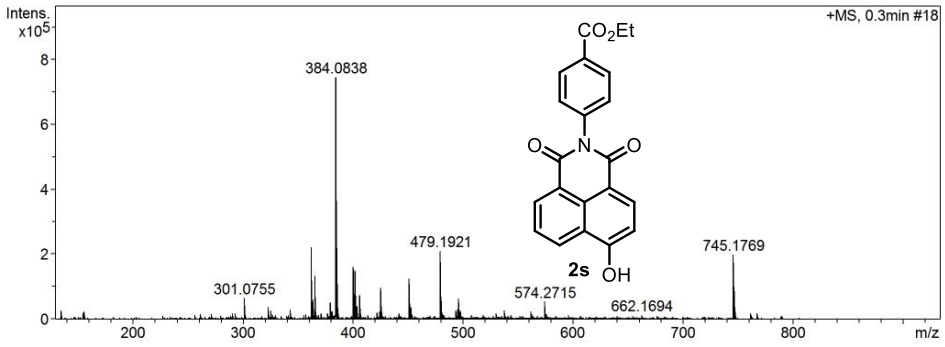
Analysis Info

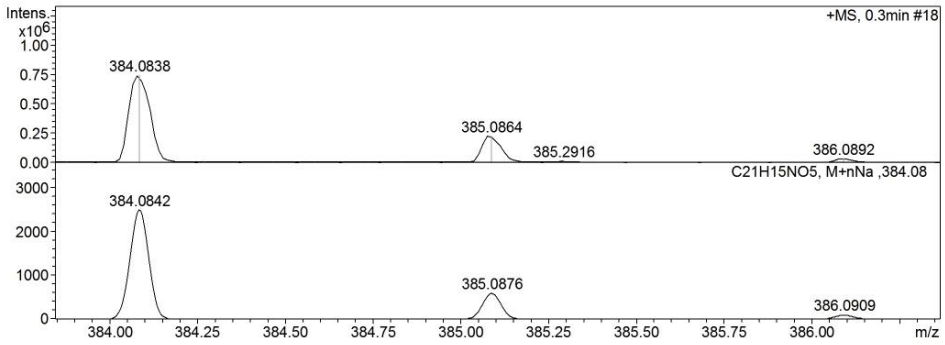
Analysis Name	D:\Data\USER DATA 2023\Jan\30-01-2023\Dr.M.Kapur-TC_03_47.d	Acquisition Date	30-01-2023 15:12:42
Method	tune_low_Jan23.m	Operator	Bruker
Sample Name	TC_03_47	Instrument	micrOTOF-Q 10330
Comment			

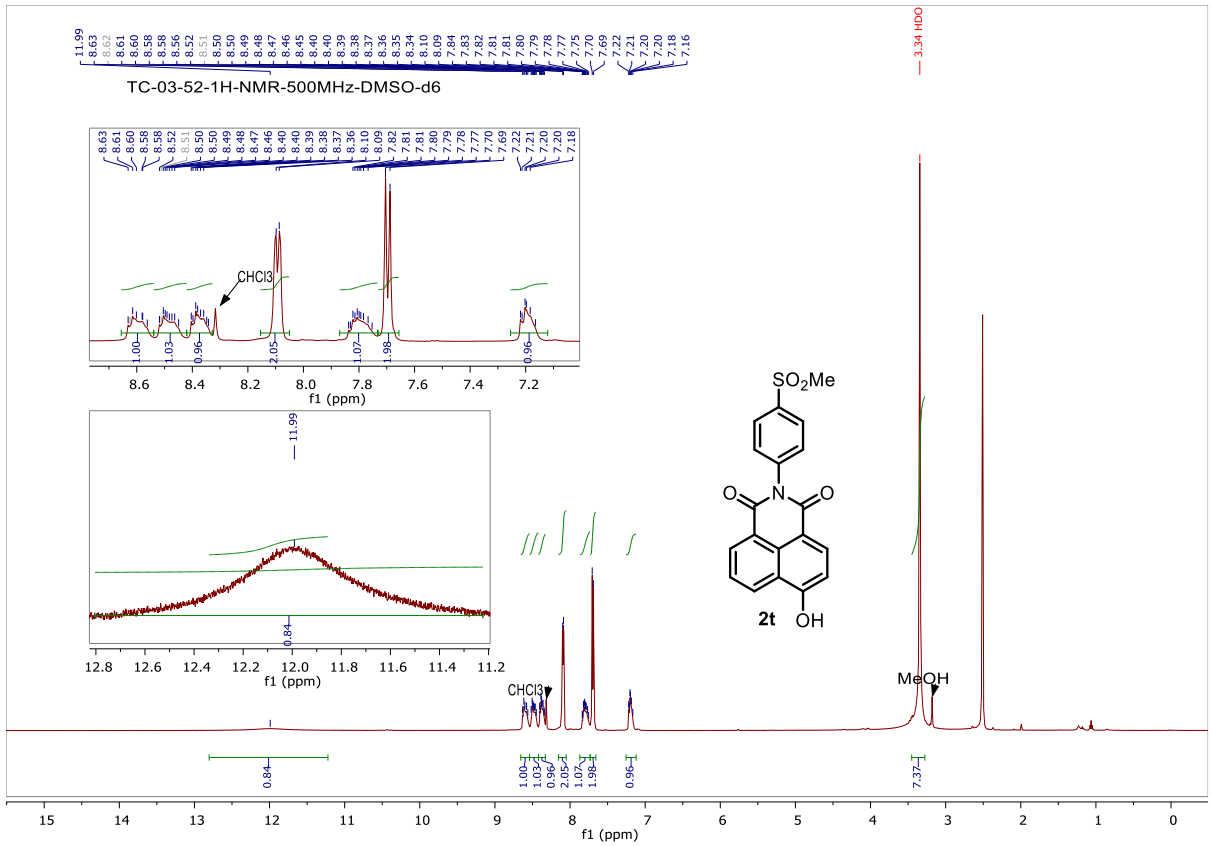
Acquisition Parameter

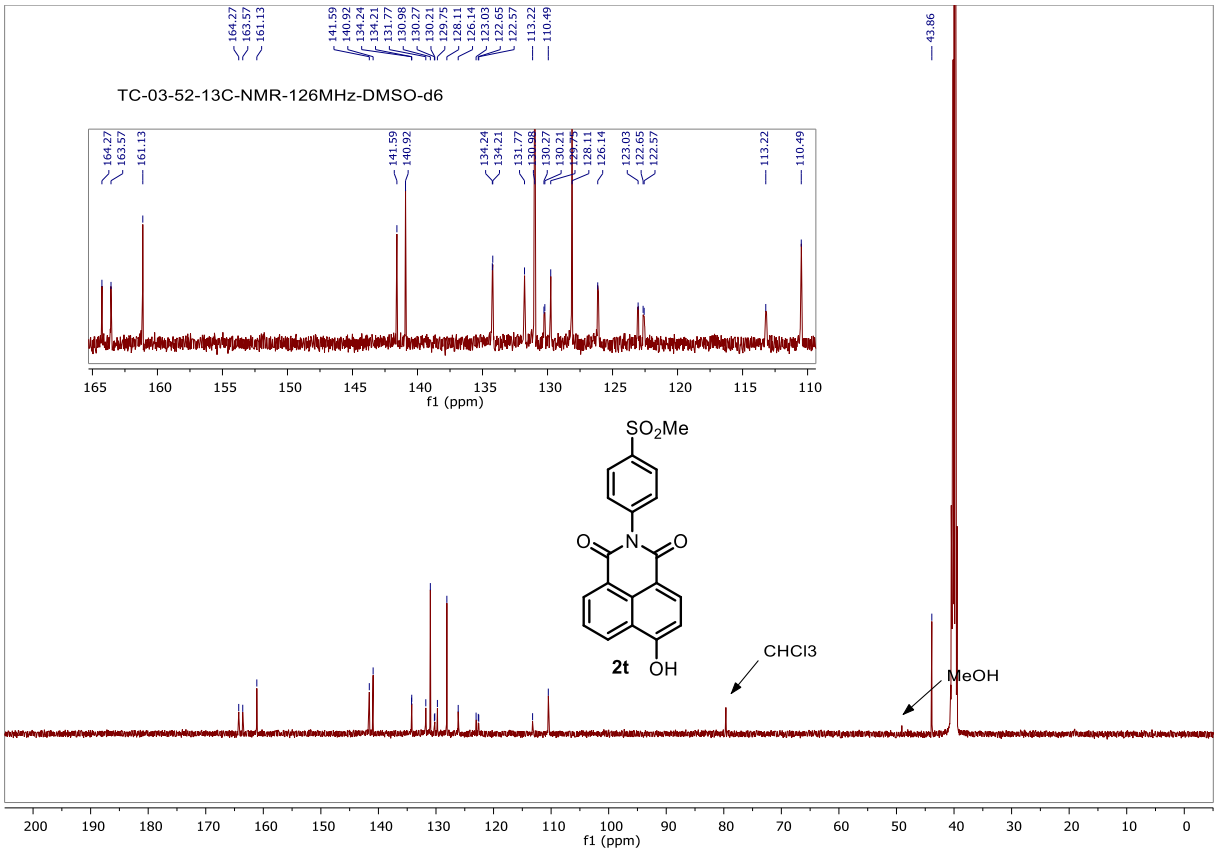
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste








TC-03-52-1H-NMR-500MHz-DMSO-d6
CHCl3
MeOH
SO2Me
2t
OH
f1 (ppm)


TC-03-52-13C-NMR-126MHz-DMSO-d6
SO2Me
2t
OH
CHCl3
MeOH
f1 (ppm)

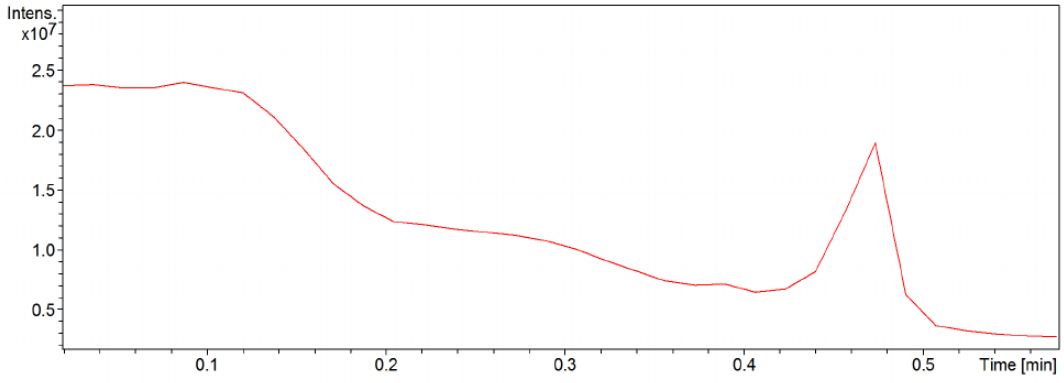
Display Report

Analysis Info

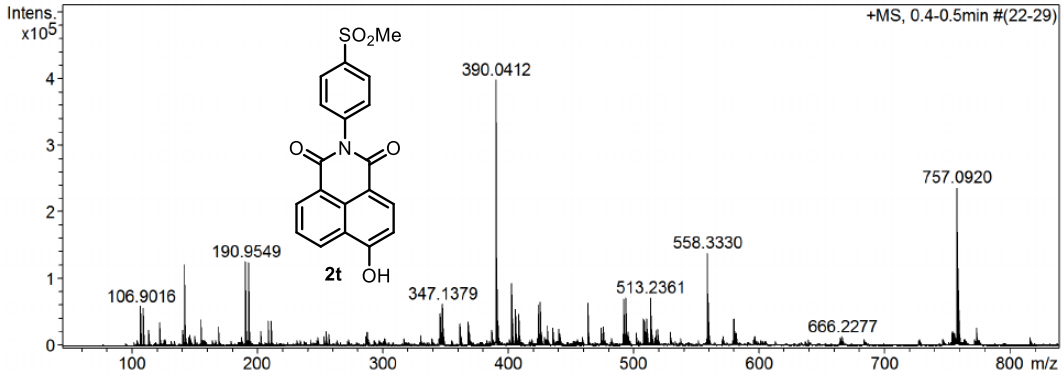
Analysis Name	D:\Data\USER DATA 2023\Feb\22-02-2023\Prof M Kapur-TC-03-52.d	Acquisition Date	22-02-2023 15:02:49
Method	tune_low_Jan23.m	Operator	Bruker
Sample Name	TC-03-52	Instrument	micrOTOF-Q 10330
Comment			

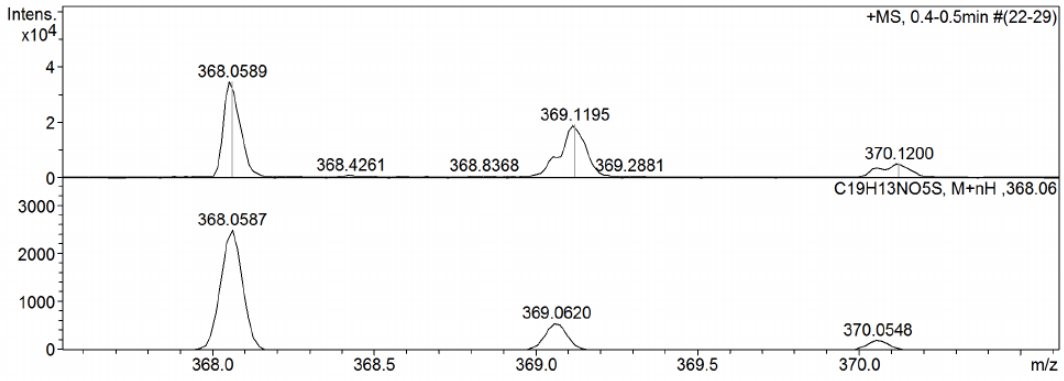
Acquisition Parameter

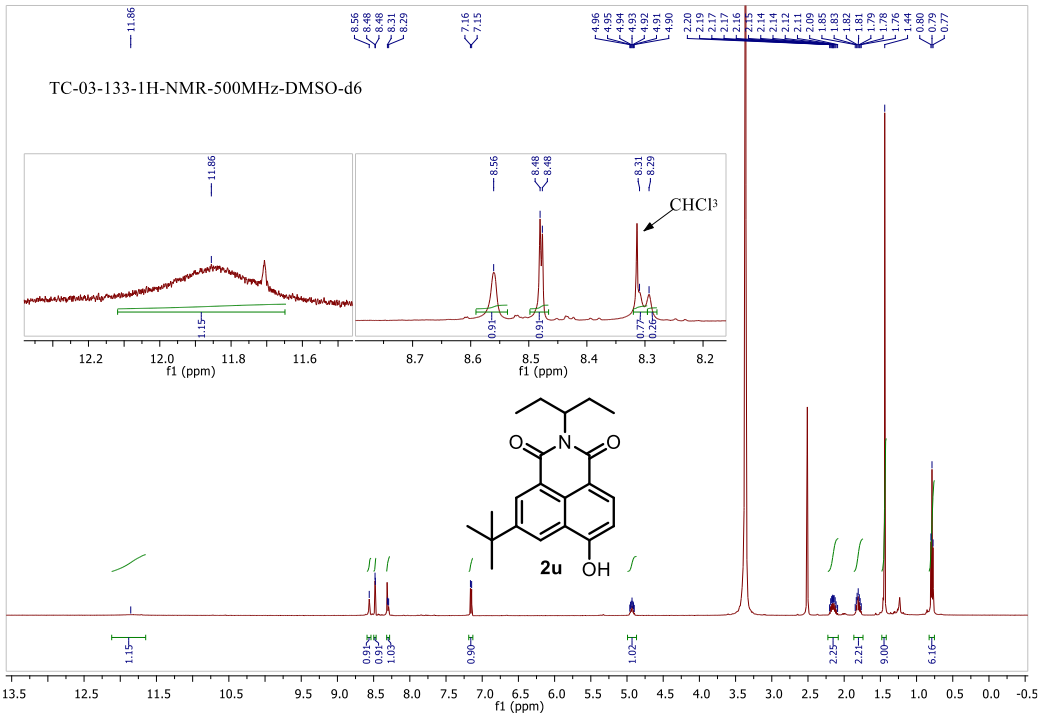
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.8 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

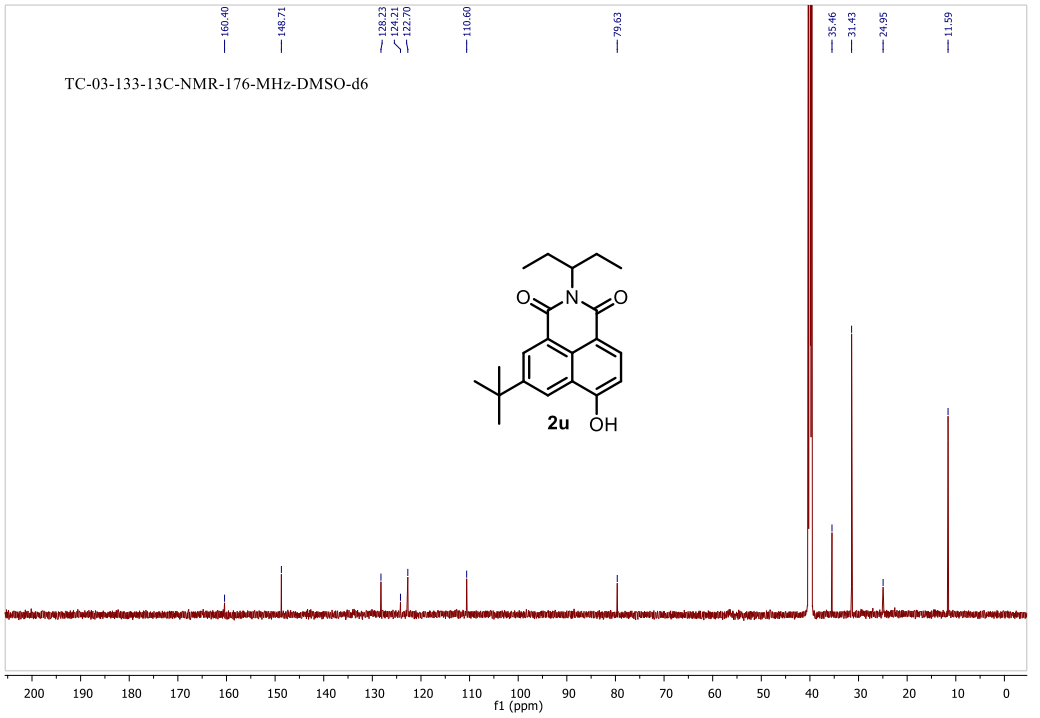


TIC +All MS






TC-03-133-1H-NMR-500MHz-DMSO-d6
CHCl3
2u
OH
f1 (ppm)


TC-03-133-13C-NMR-176-MHz-DMSO-d6
2u
OH
f1 (ppm)

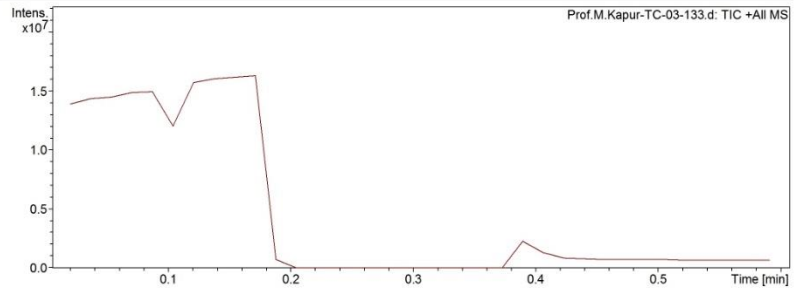
Display Report

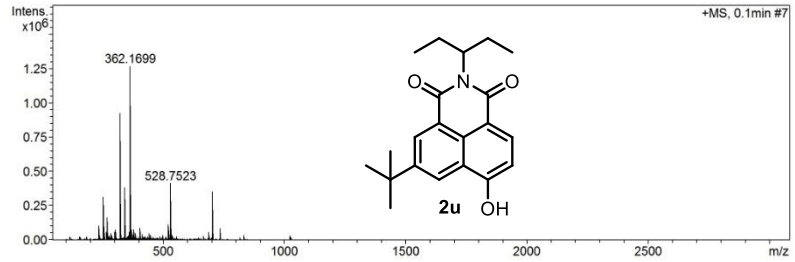
Analysis Info

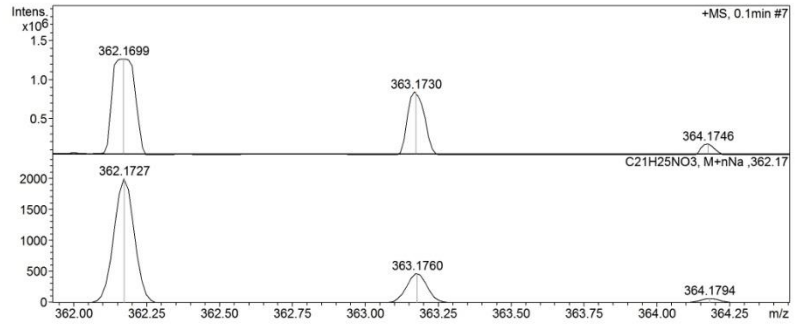
Analysis Name	D:\Data\USER DATA 2024\01-JAN\Prof.M.Kapur-TC-03-133.d	Acquisition Date	01-01-2024 14:56:19
Method	tune_low_APR23.m	Operator	Bruker
Sample Name	TC-03-133	Instrument	micrOTOF-Q 10330
Comment			

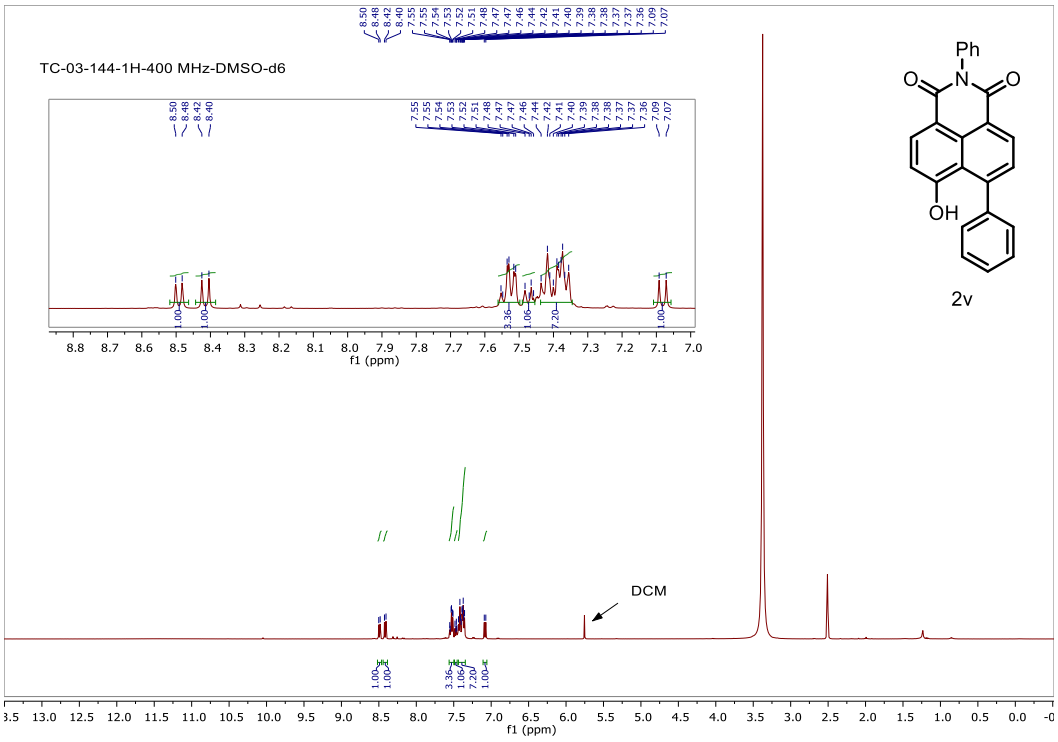
Acquisition Parameter

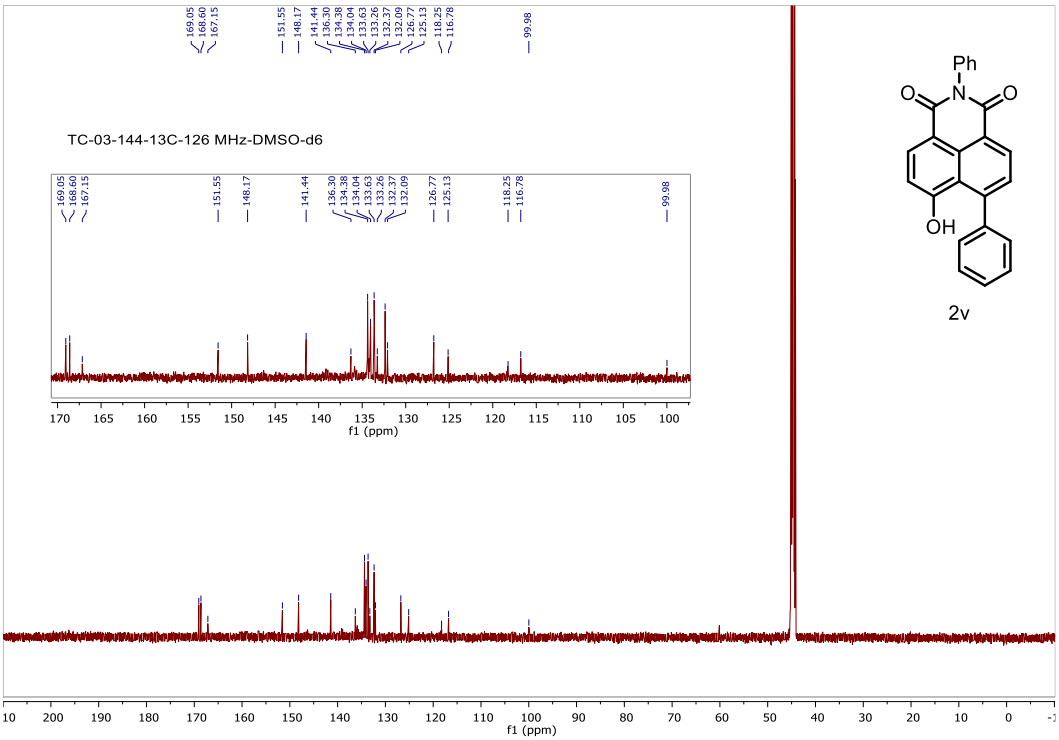
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste








TC-03-144-1H-400 MHz-DMSO-d6
Ph
O
N
O
OH
2v
DCM
f1 (ppm)


TC-03-144-13C-126 MHz-DMSO-d6
Ph
O
N
O
OH
2v
f1 (ppm)

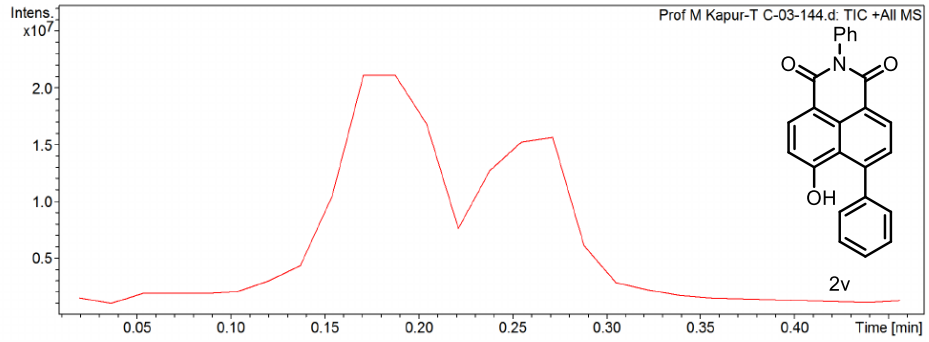
Display Report

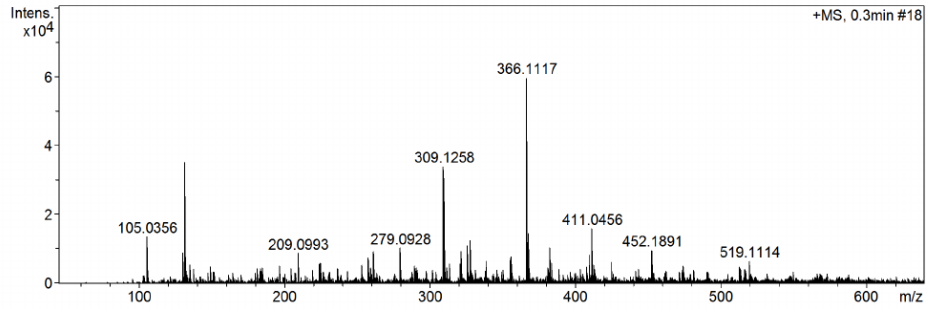
Analysis Info

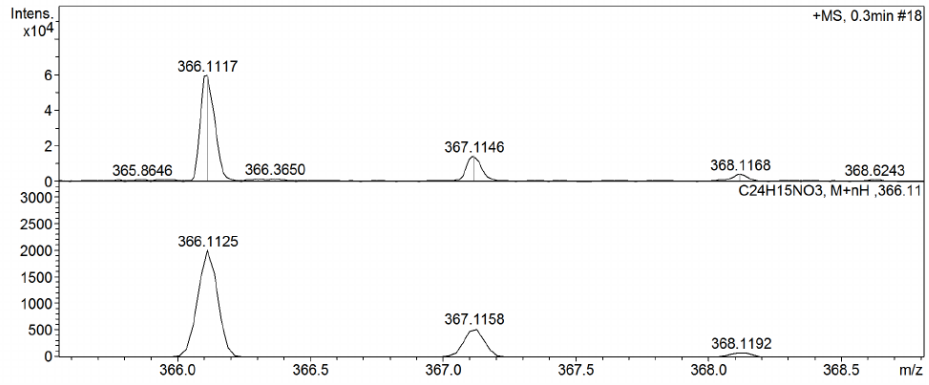
Analysis Name	D:\Data\USER DATA 2024\08-jan\Prof M Kapur-T C-03-144.d	Acquisition Date	08-01-2024 16:32:47
Method	tune_low_APR23.m	Operator	Bruker
Sample Name	TC-03-144	Instrument	micrOTOF-Q 10330
Comment			

Acquisition Parameter

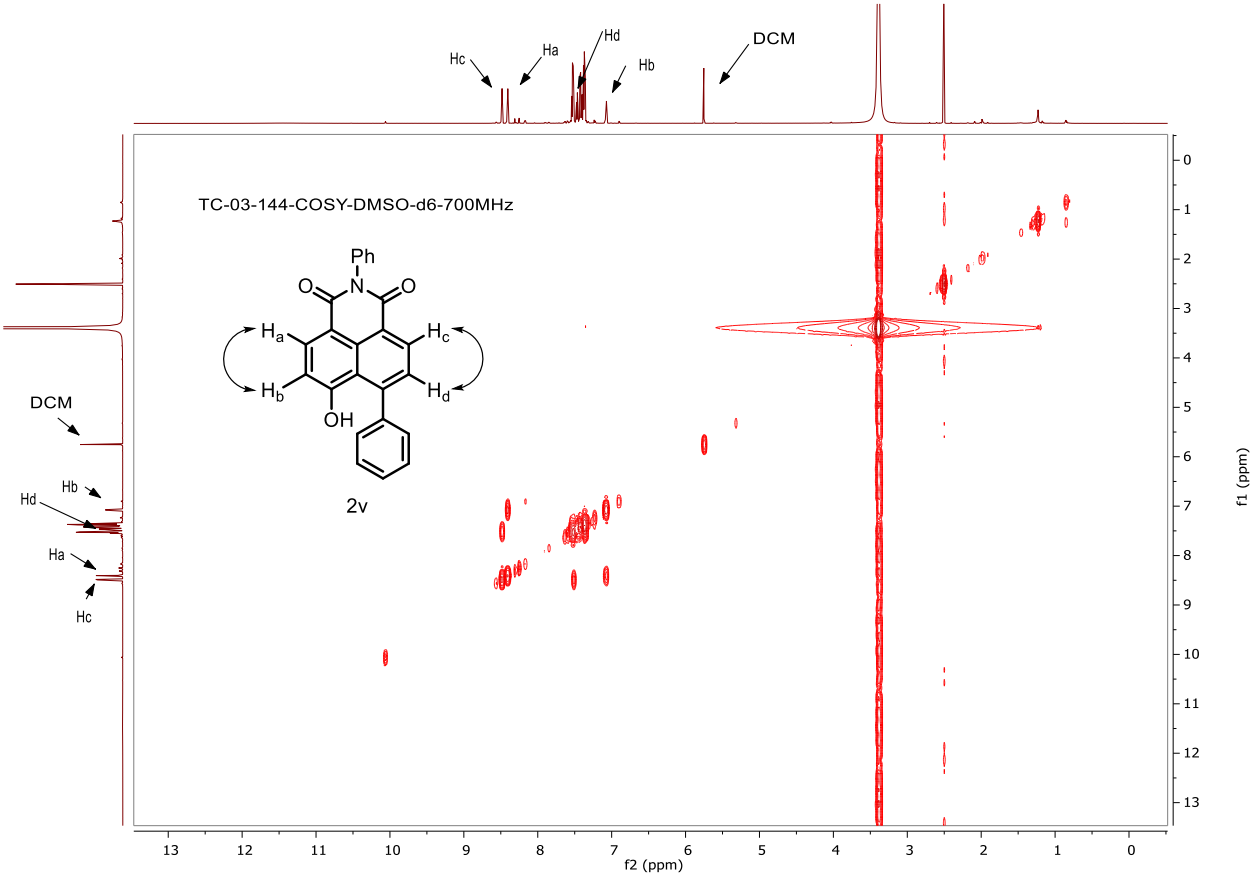
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

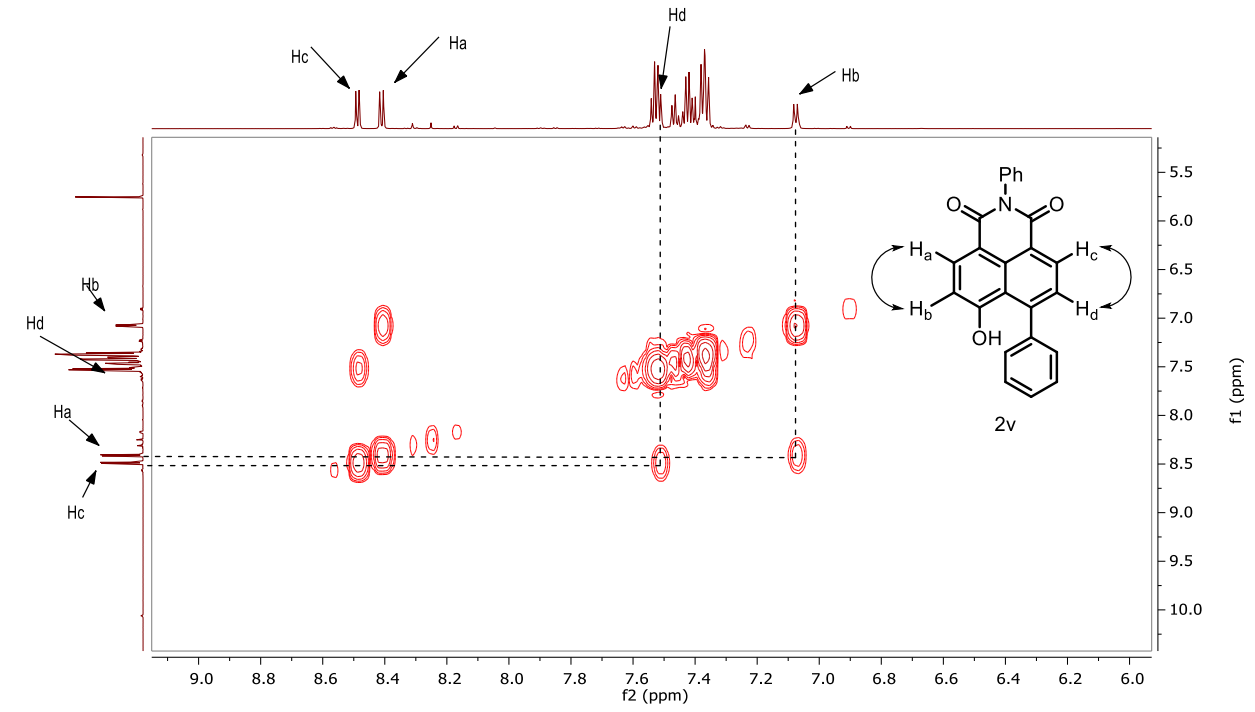


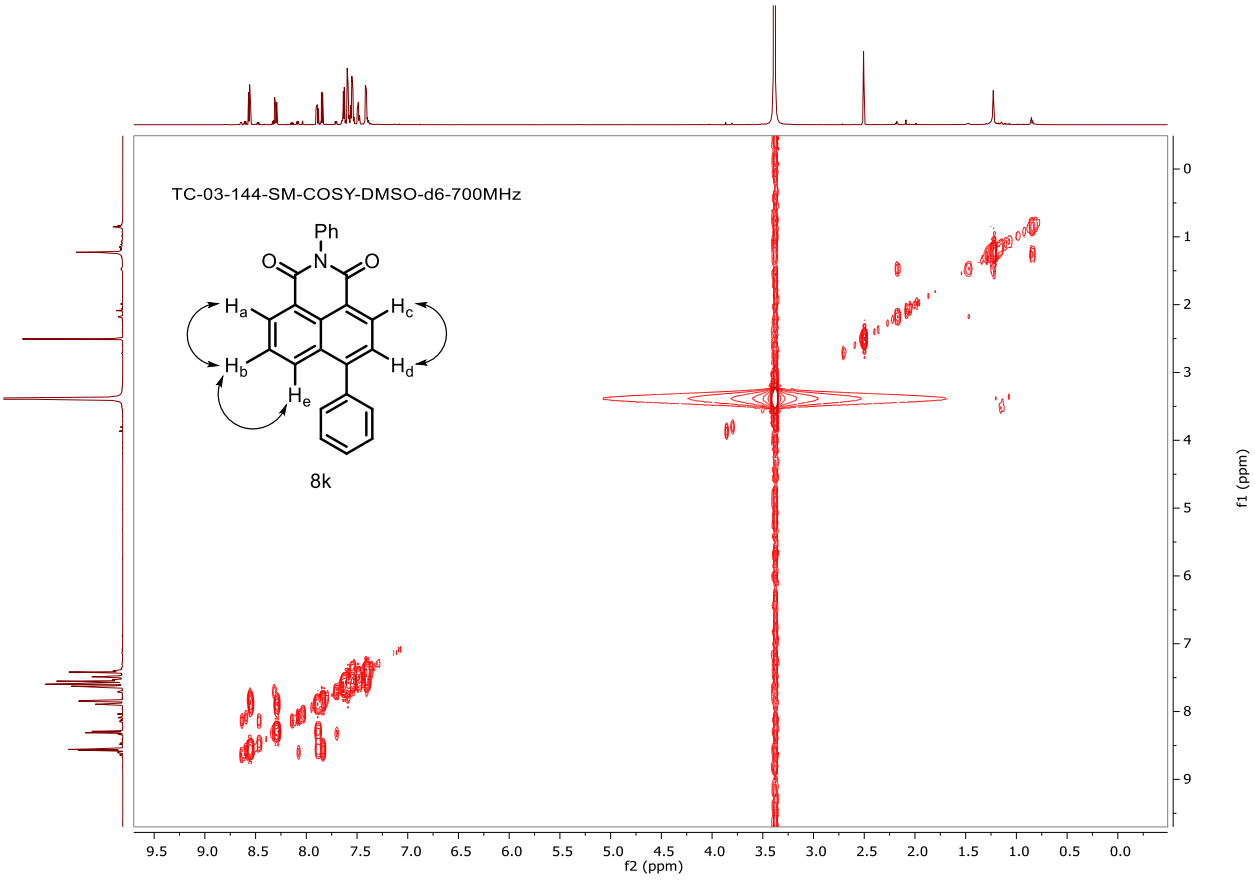


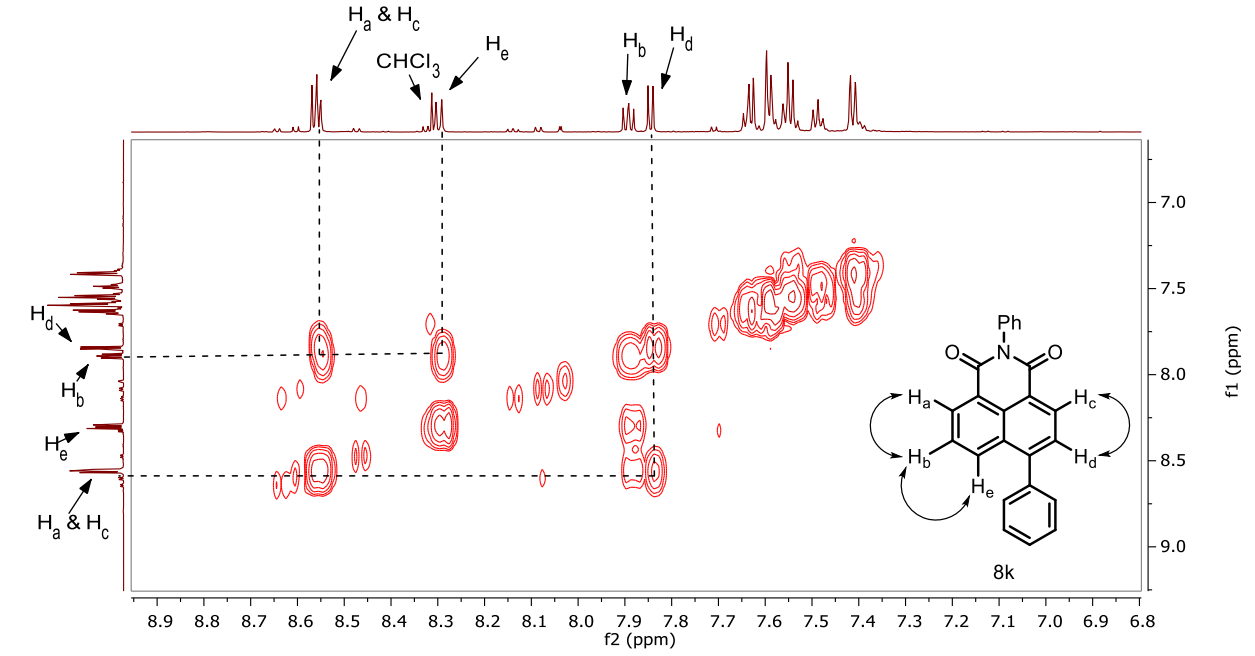


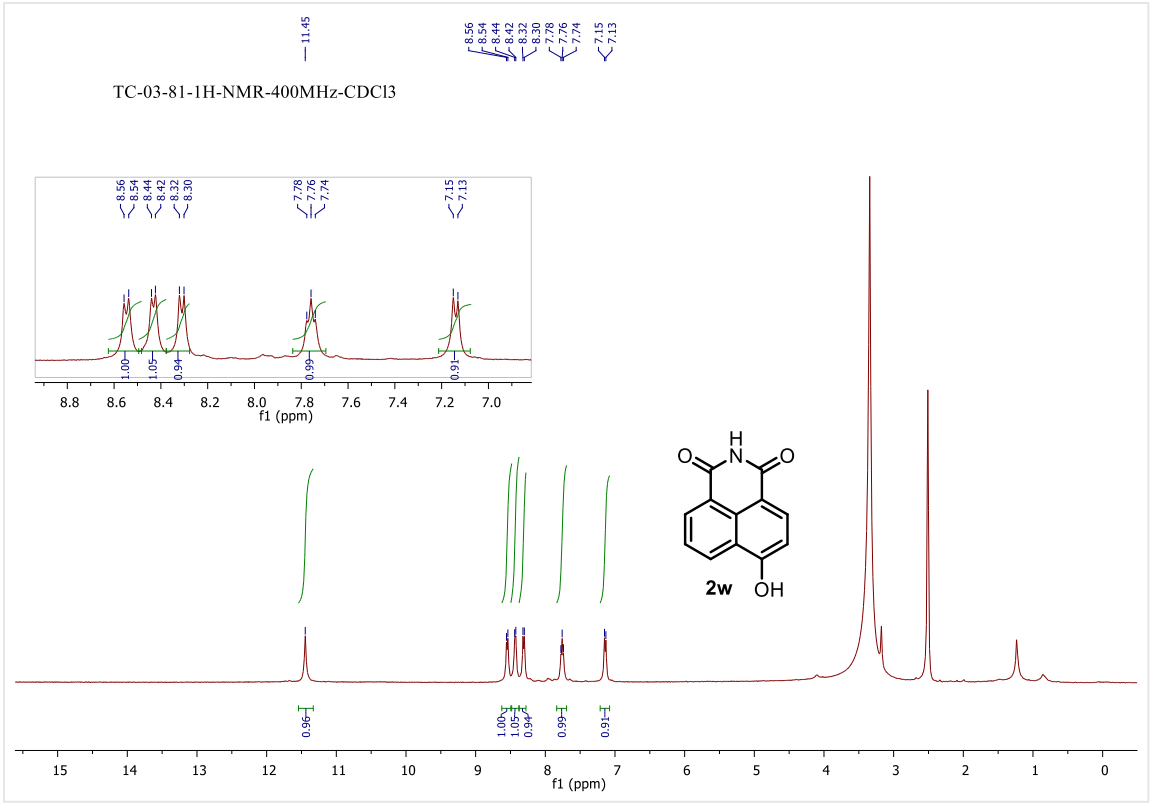
NMR Study of Regioselectivity (2v):

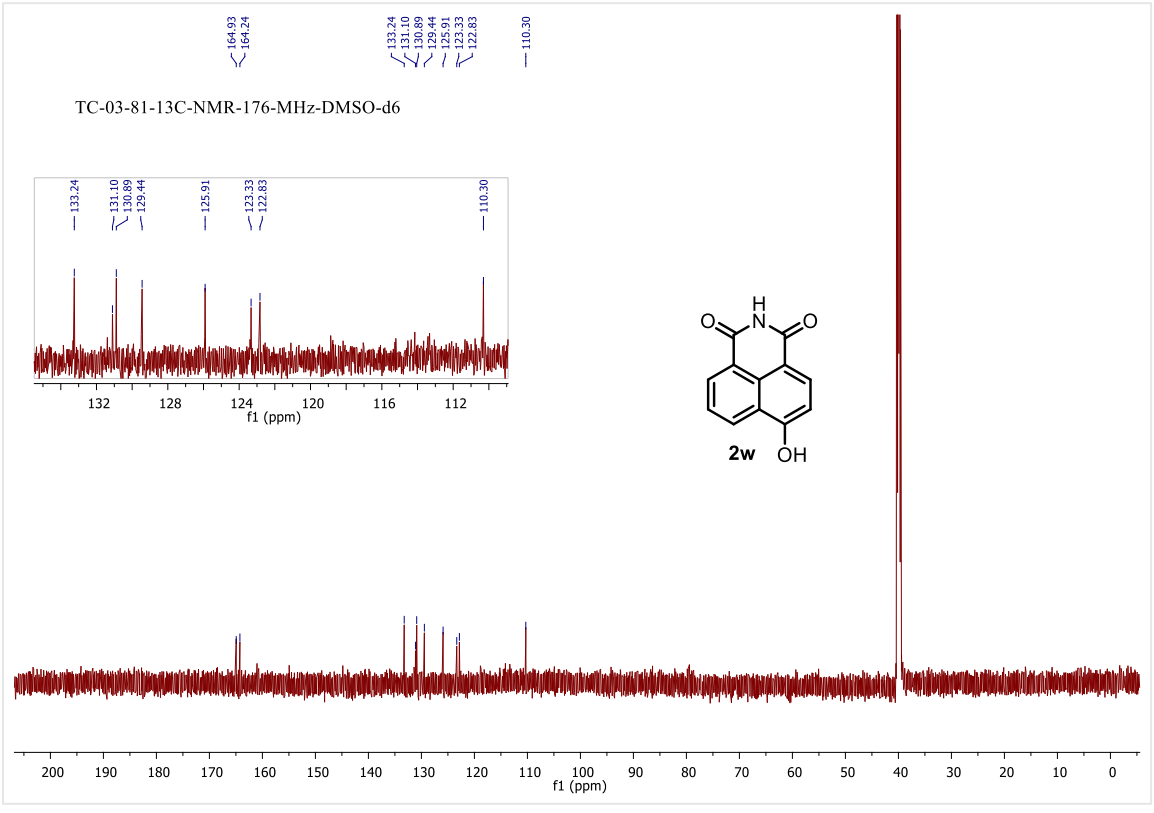





TC-03-144-SM-COSY-DMSO-d6-700MHz
Ph
O
N
O
Ha
Hb
Hc
Hd
He
8k
f2 (ppm)
f1 (ppm)


Ha & Hc
CHCl3
He
Hb
Hd
Hd
Hb
He
Ha & Hc
Ph
O
N
O
Ha
Hb
Hc
Hd
He
8k
f2 (ppm)
f1 (ppm)


TC-03-81-1H-NMR-400MHz-CDCl3
11.45
8.56
8.54
8.44
8.42
8.32
8.30
7.78
7.76
7.74
7.15
7.13
f1 (ppm)
2w
OH


TC-03-81-13C-NMR-176-MHz-DMSO-d6
164.93
164.24
133.24
131.10
130.89
129.44
125.91
123.33
122.83
110.30
f1 (ppm)
2w
OH

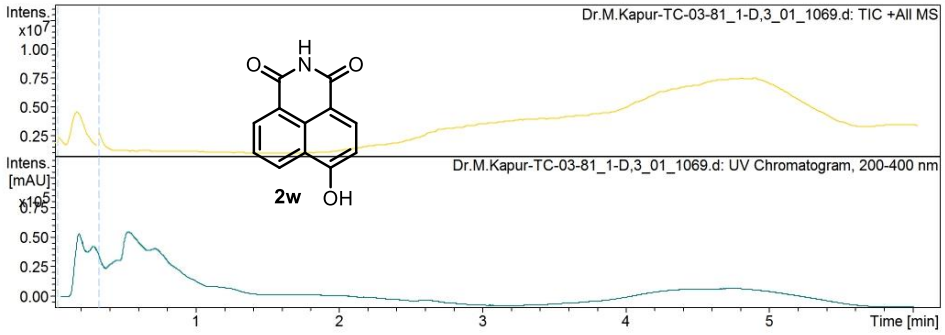
Display Report

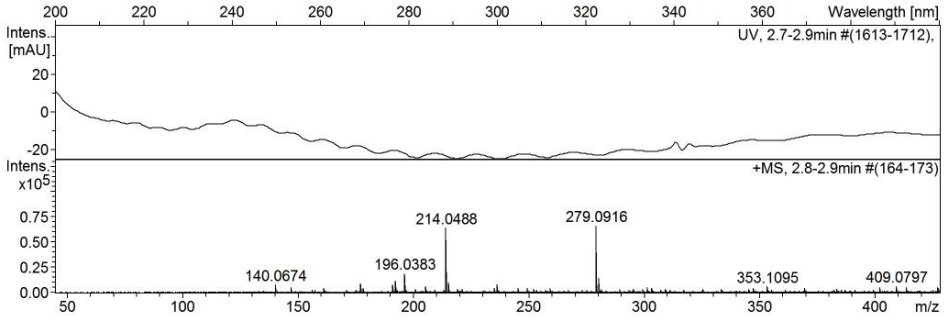
Analysis Info

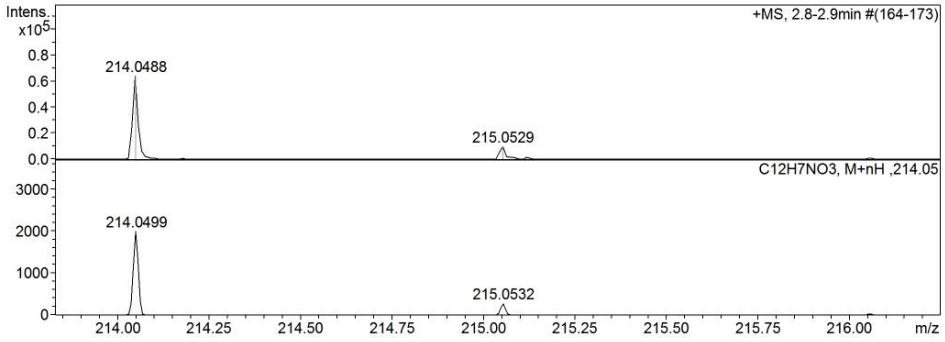
Analysis Name	D:\Data\USER DATA 2023\may\08-May\Dr.M.Kapur-TC-03-81_1-D,3_01_1069.d	Acquisition Date	08-05-2023 14:34:16
Method	HRLCMS-20 APR23.m	Operator	Bruker
Sample Name	Dr.M.Kapur-TC-03-81	Instrument	micrOTOF-Q 10330
Comment			

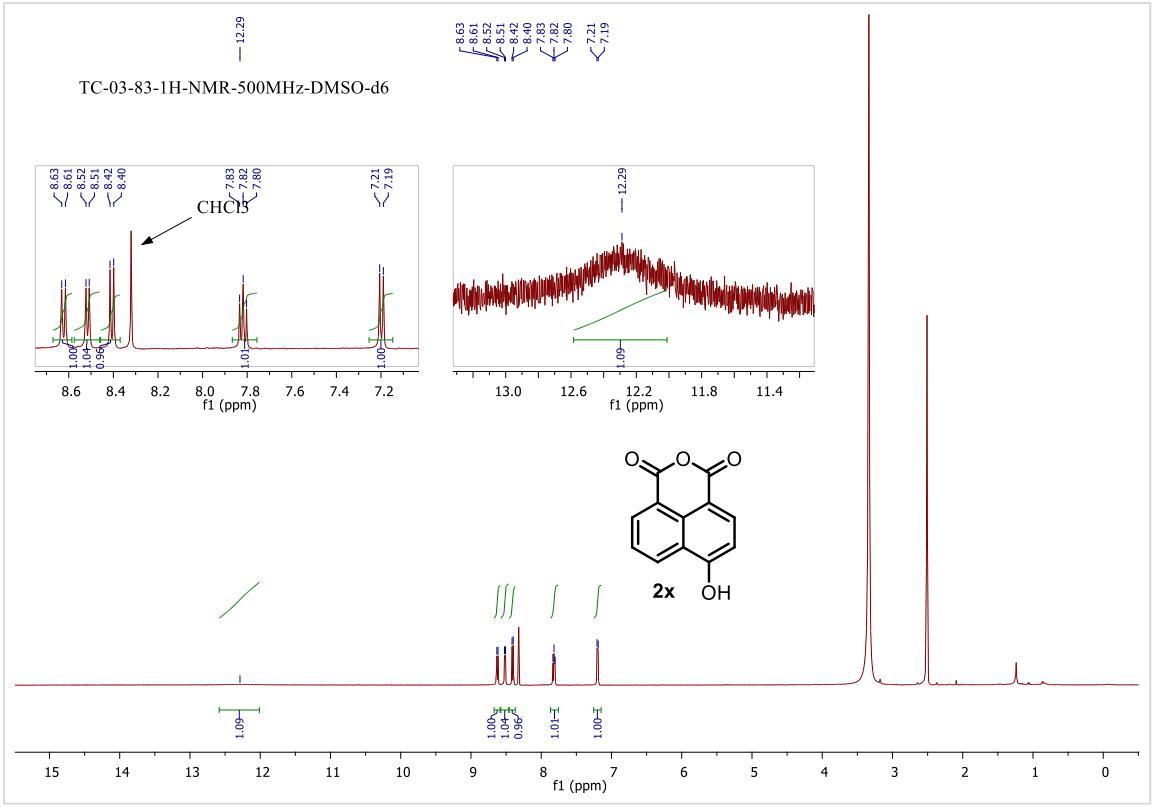
Acquisition Parameter

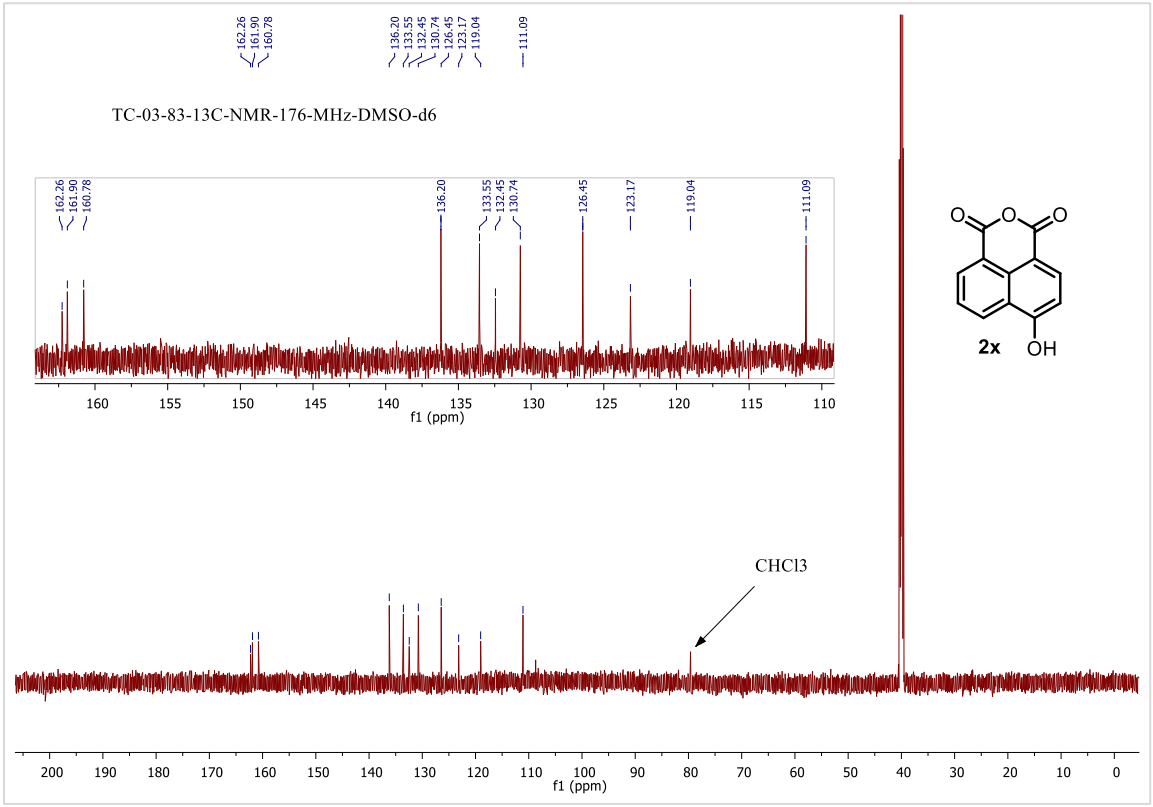
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste








TC-03-83-1H-NMR-500MHz-DMSO-d6
CHCl3
2x
OH
f1 (ppm)


TC-03-83-13C-NMR-176-MHz-DMSO-d6
CHCl3
2x
OH
f1 (ppm)

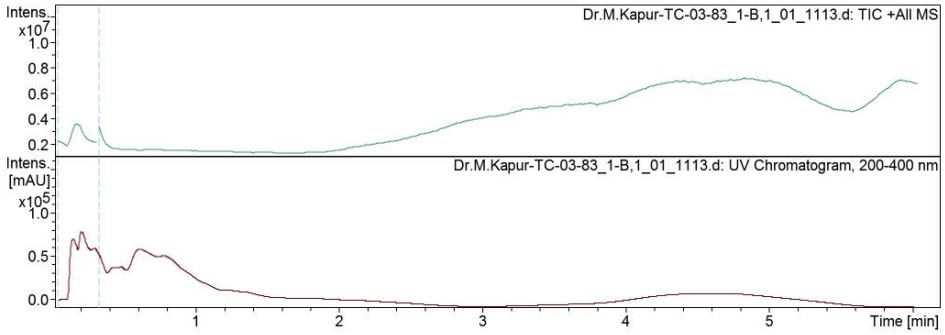
Display Report

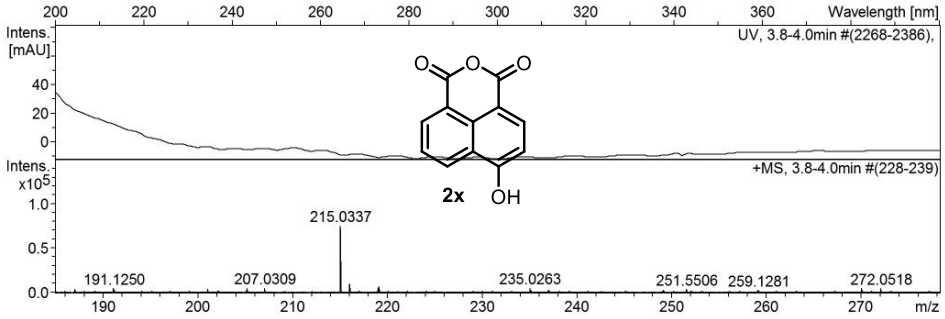
Analysis Info

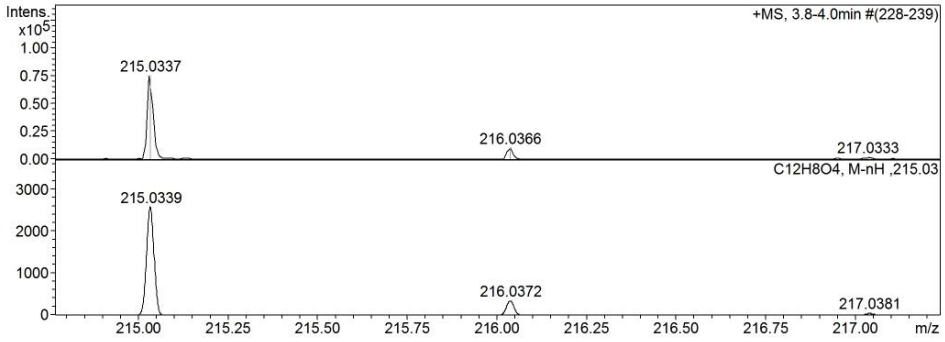
Analysis Name	D:\Data\USER DATA 2023\may\09-may\Dr.M.Kapur-TC-03-83_1-B,1_01_1113.d	Acquisition Date	09-05-2023 14:29:45
Method	HRLCMS-20 APR23.m	Operator	Bruker
Sample Name	Dr.M.Kapur-TC-03-83	Instrument	micrOTOF-Q 10330
Comment			

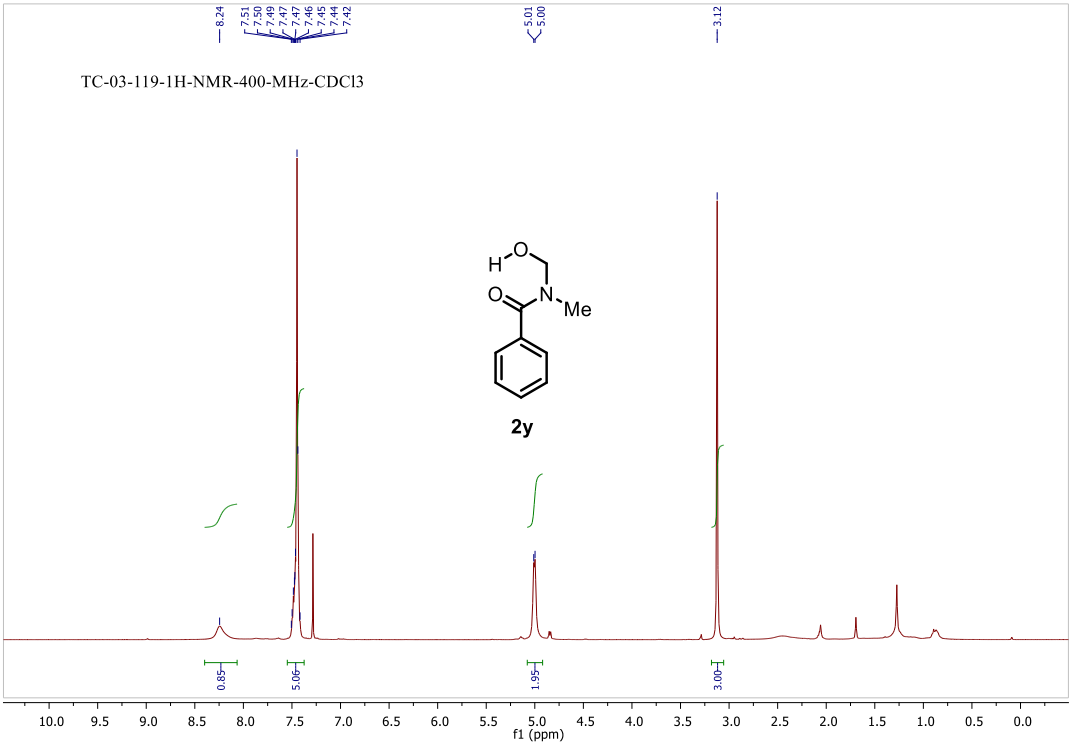
Acquisition Parameter

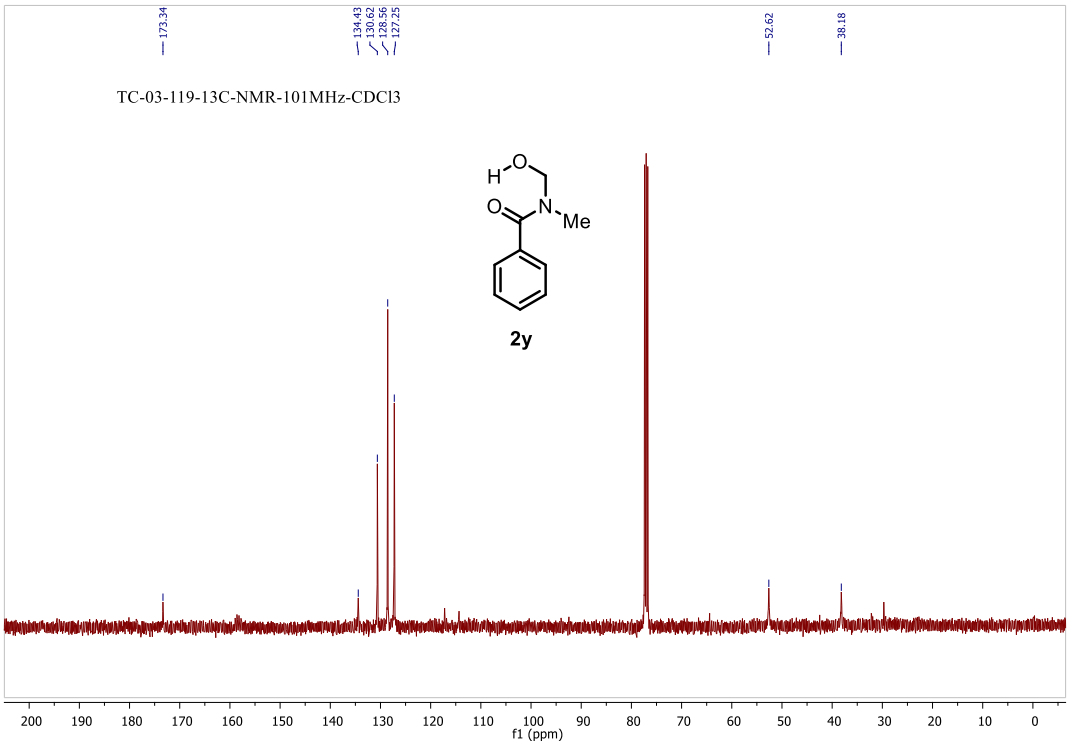
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste








8.24
7.51
7.50
7.49
7.47
7.47
7.46
7.45
7.44
7.42
5.01
5.00
3.12
TC-03-119-1H-NMR-400-MHz-CDCl3
H
O
O
N
Me
2y
0.85
5.06
1.95
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


173.34
134.43
130.62
128.56
127.25
52.62
38.18
TC-03-119-13C-NMR-101MHz-CDCl3
H
O
O
N
Me
2y
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

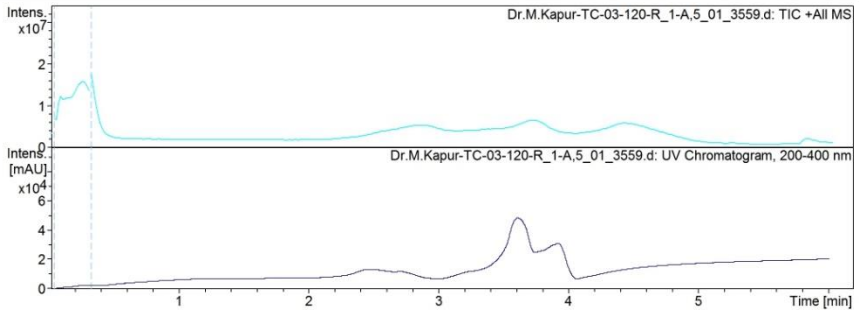
Display Report

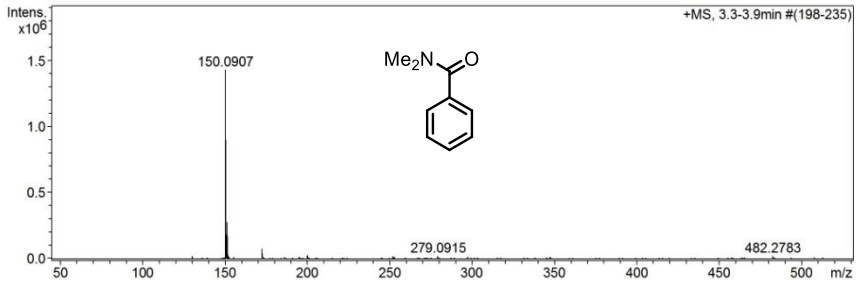
Analysis Info

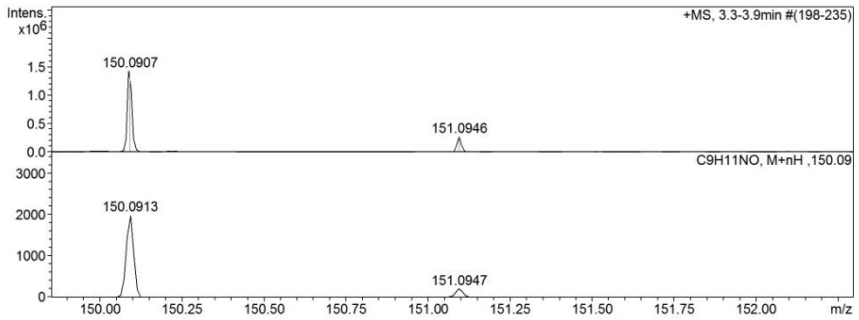
Analysis Name	D:\Data\USER DATA 2023\Dec23\01-Dec\Dr.M.Kapur-TC-03-120-R_1-A,5_01_3559.d	Acquisition Date	01-12-2023 11:40:56
Method	HRLCMS-20 Sept.m	Operator	Bruker
Sample Name	Dr.M.Kapur-TC-03-120-R	Instrument	micrOTOF-Q 10330
Comment			

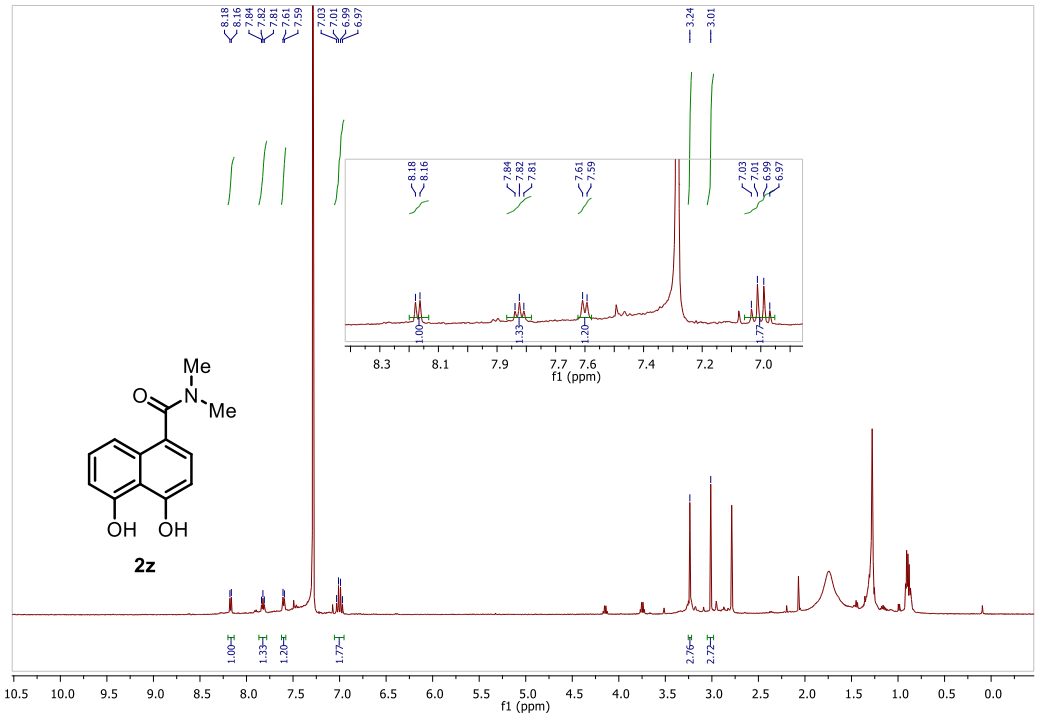
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste








8.18
8.16
7.84
7.82
7.81
7.61
7.59
7.03
7.01
6.99
6.97
3.24
3.01
Me
N
Me
O
OH
OH
2z
1.00
1.33
1.20
1.77
2.76
2.72
f1 (ppm)

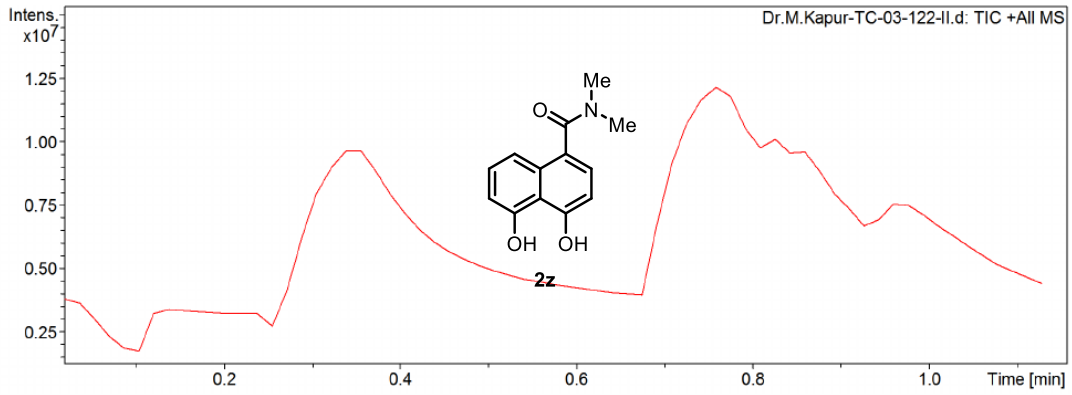
Display Report

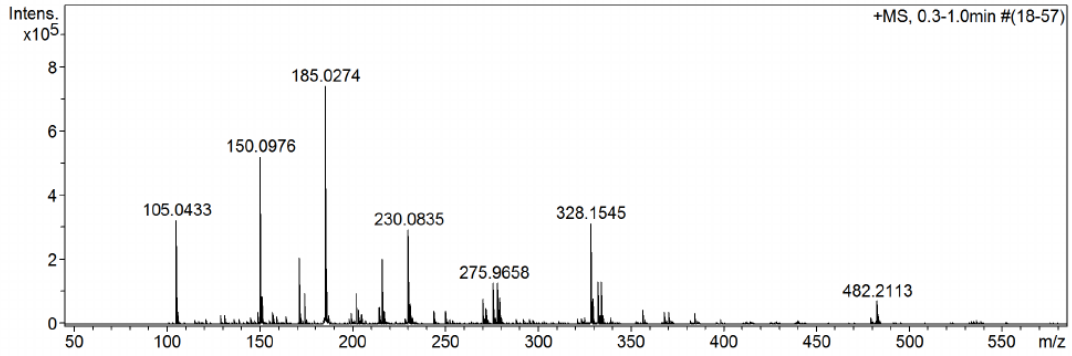
Analysis Info

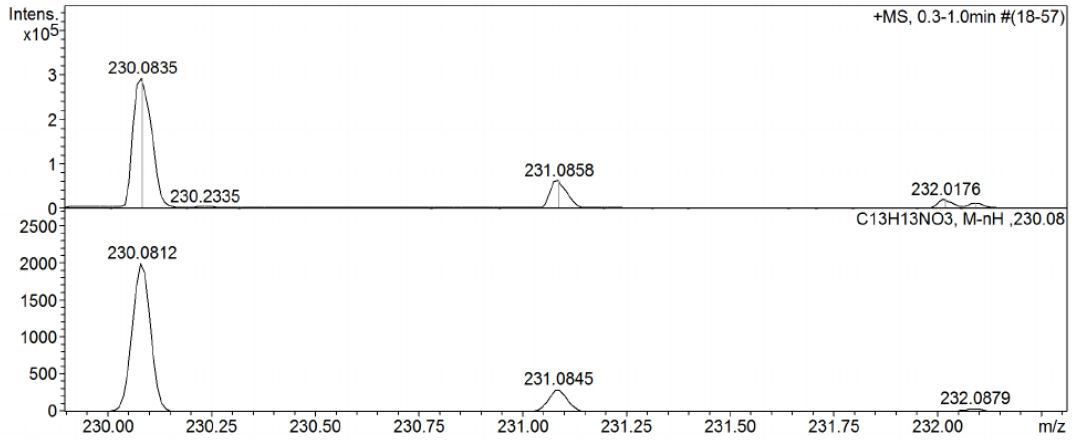
Analysis Name	D:\Data\USER DATA 2023\Nov23\30-nov\Dr.M.Kapur-TC-03-122-II.d	Acquisition Date	30-11-2023 14:34:12
Method	tune_low_APCI.m	Operator	Bruker
Sample Name	TC-03-122-II	Instrument	micrOTOF-Q 10330
Comment			

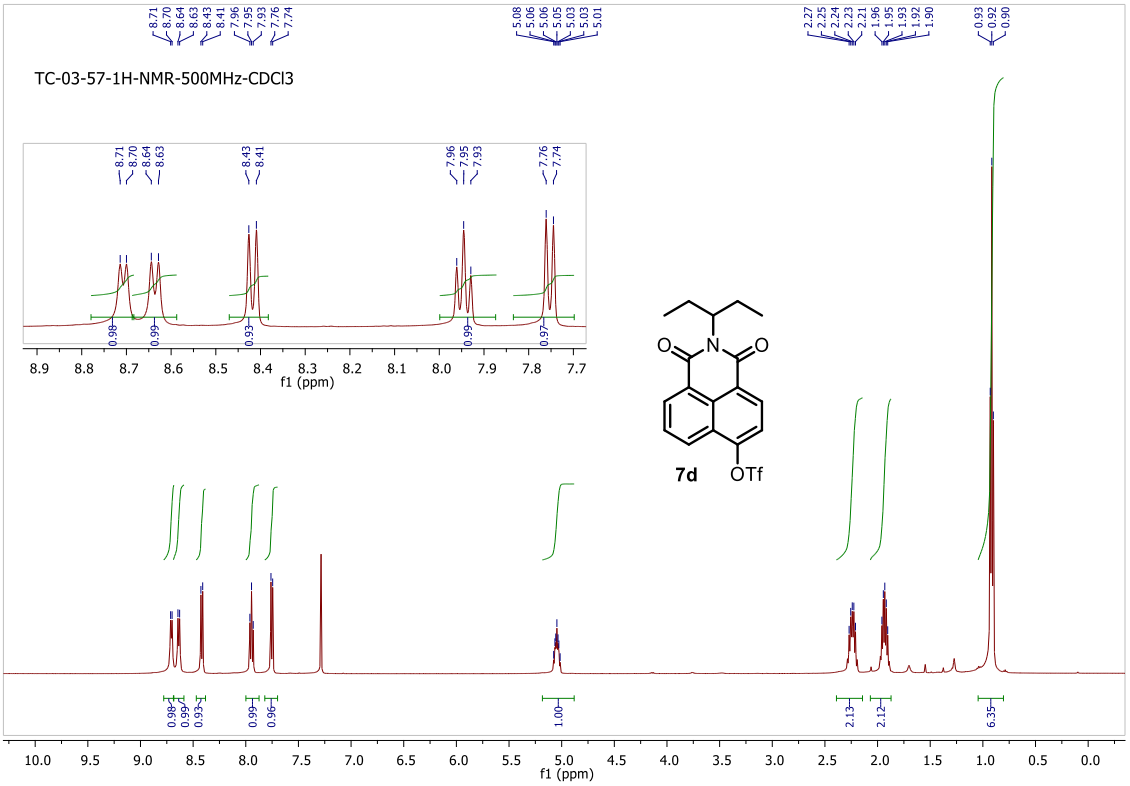
Acquisition Parameter

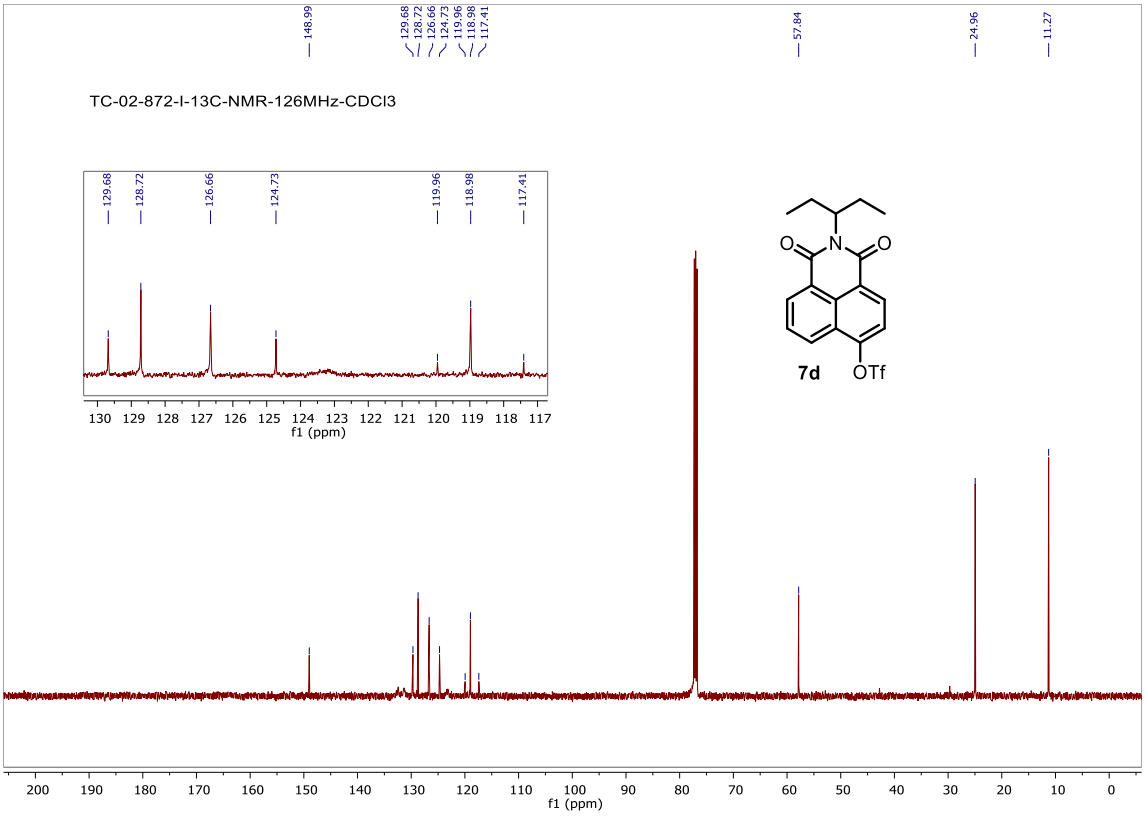
Source Type	Multi Mode	Ion Polarity	Positive	Set Nebulizer	2.0 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	5.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

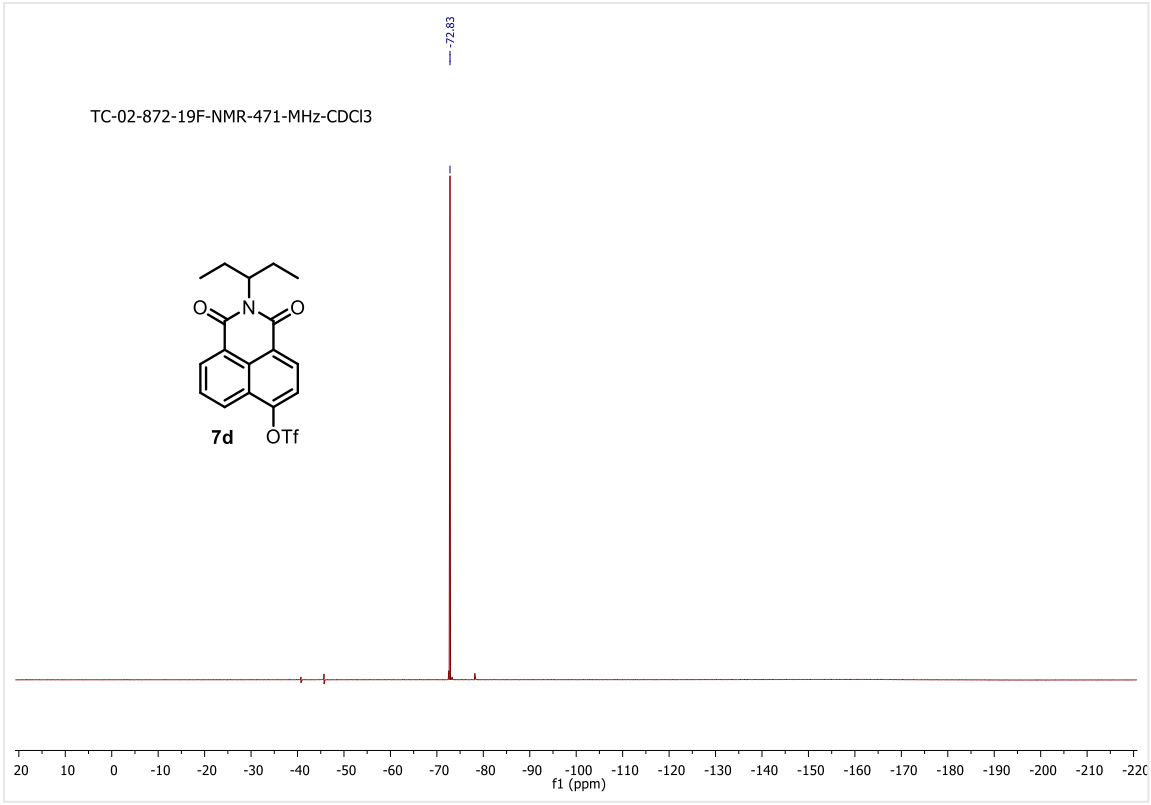







TC-03-57-1H-NMR-500MHz-CDCl3
7d
OTf
f1 (ppm)


TC-02-872-I-13C-NMR-126MHz-CDCl3
7d
OTf
f1 (ppm)


-72.83
TC-02-872-19F-NMR-471-MHz-CDCl3
O
N
O
7d
OTf
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

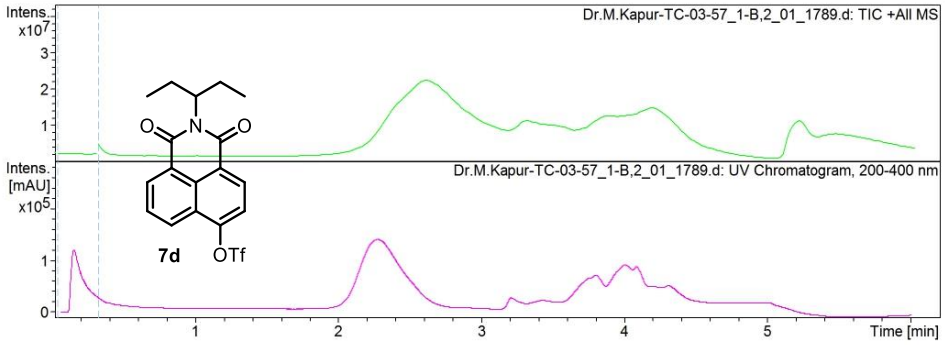
Display Report

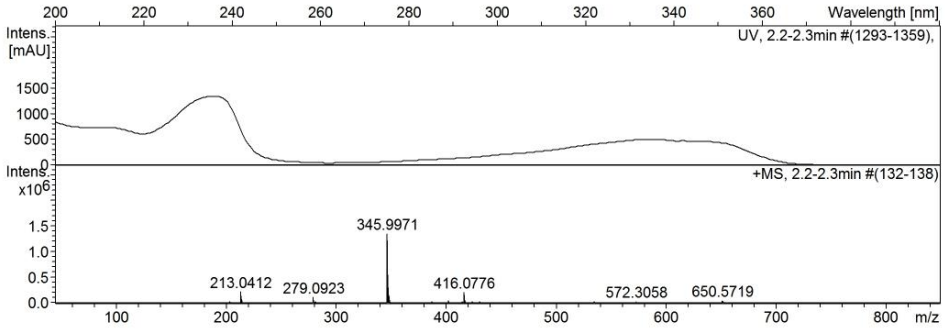
Analysis Info

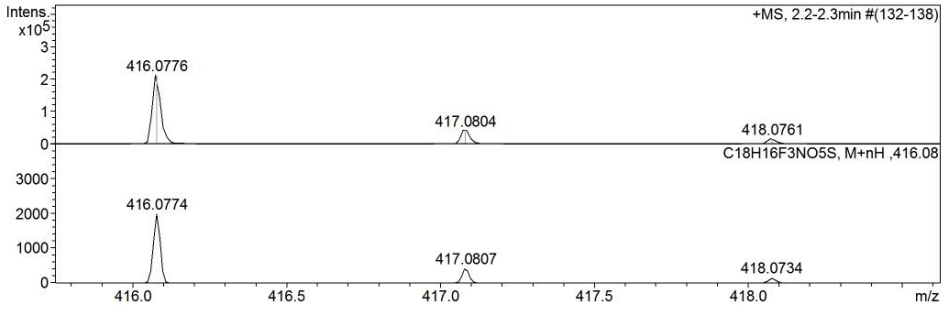
Analysis Name	D:\Data\USER DATA 2023\June\09-june\Dr.M.Kapur-TC-03-57_1-B,2_01_1789.d	Acquisition Date	09-06-2023 13:05:09
Method	HRLCMS-20 APR23.m	Operator	Bruker
Sample Name	Dr.M.Kapur-TC-03-57	Instrument	micrOTOF-Q 10330
Comment			

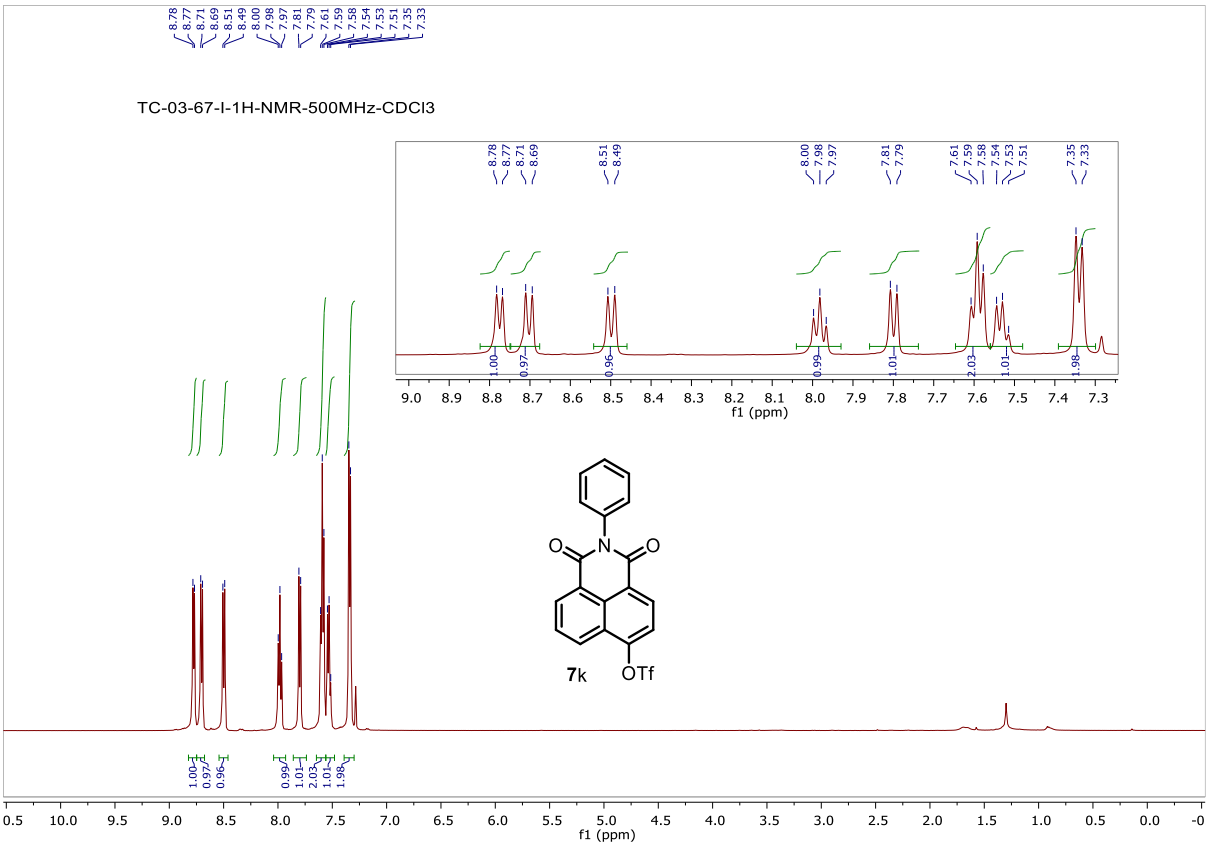
Acquisition Parameter

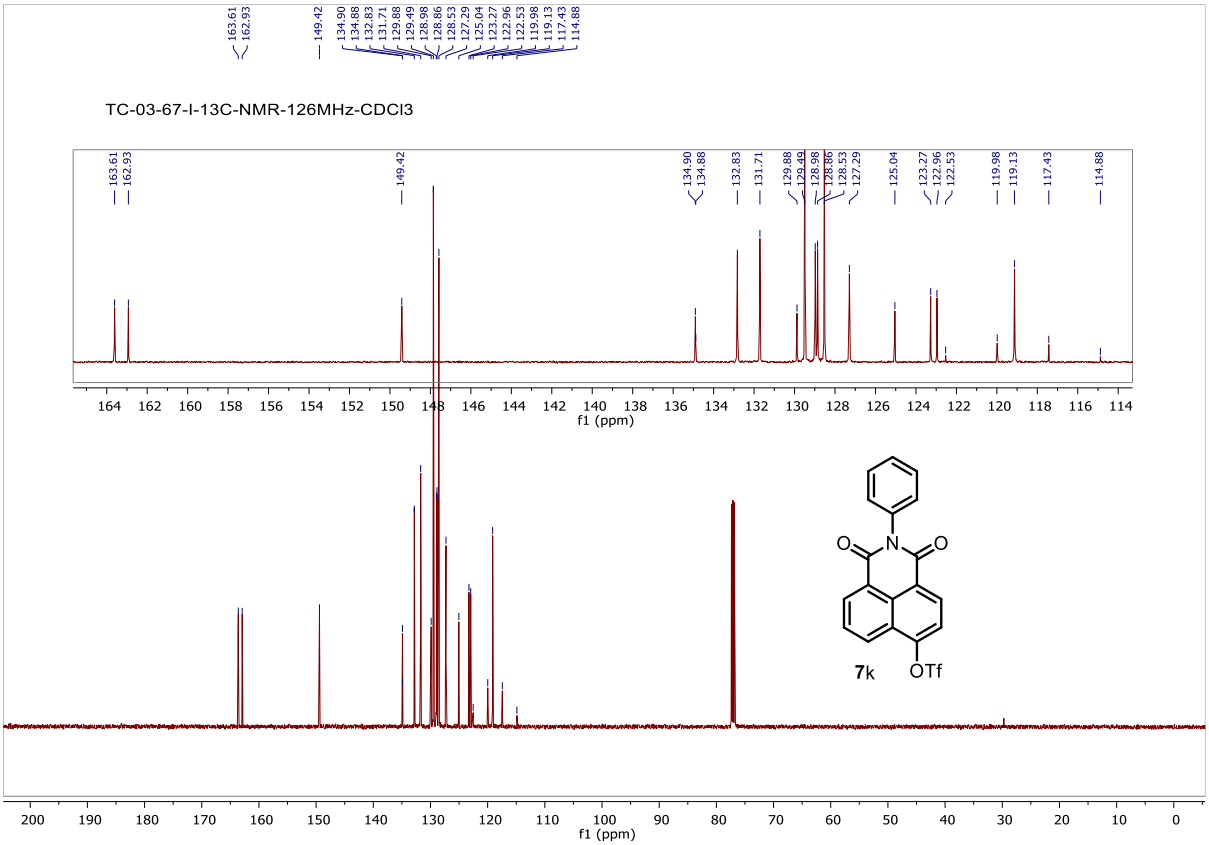
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

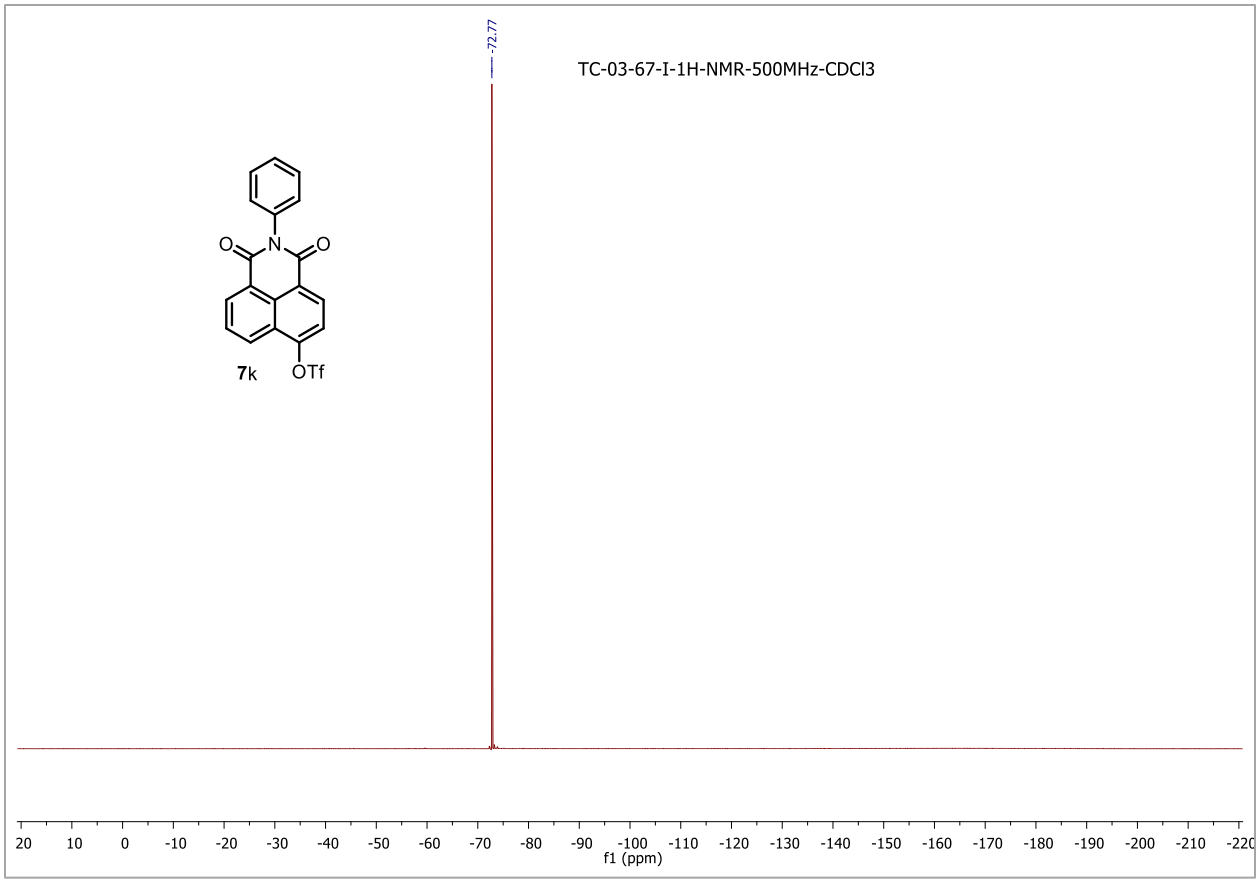







TC-03-67-I-1H-NMR-500MHz-CDCl3
8.78
8.77
8.71
8.69
8.51
8.49
8.00
7.98
7.97
7.81
7.79
7.61
7.59
7.58
7.54
7.53
7.51
7.35
7.33
1.00
0.97
0.96
0.99
1.01
2.03
1.01
1.98
f1 (ppm)
O
N
O
7k
OTf


TC-03-67-I-13C-NMR-126MHz-CDCl3
163.61
162.93
149.42
134.90
134.88
132.83
131.71
129.88
129.49
128.98
128.86
128.53
127.29
125.04
123.27
122.96
122.53
119.98
119.13
117.43
114.88
f1 (ppm)
O
N
O
7k
OTf


-72.77
TC-03-67-I-1H-NMR-500MHz-CDCl3
O
N
O
7k
OTf
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

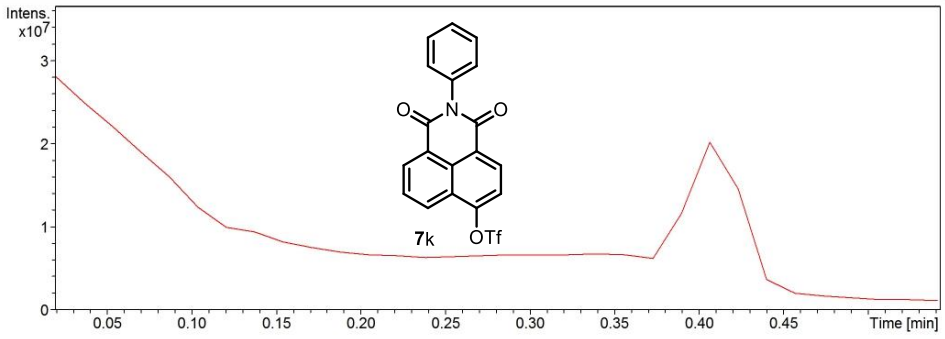
Display Report

Analysis Info

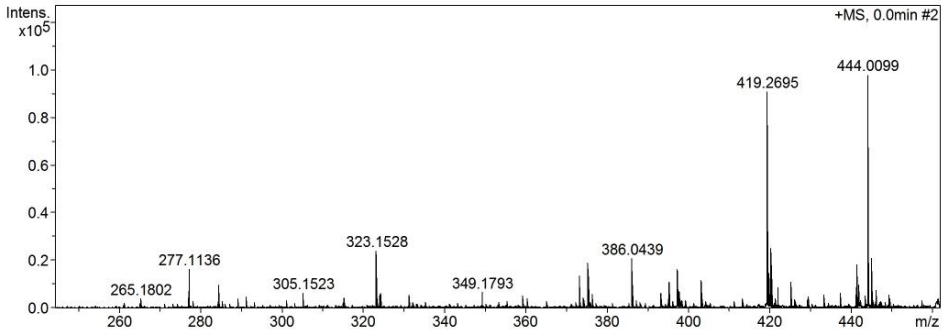
Analysis Name	D:\Data\USER DATA 2023\Feb\28-02-2023\Prof M Kapur-TC-03-67.d	Acquisition Date	28-02-2023 11:55:46
Method	tune_low_Jan23.m	Operator	Bruker
Sample Name	TC-03-67	Instrument	micrOTOF-Q 10330
Comment			

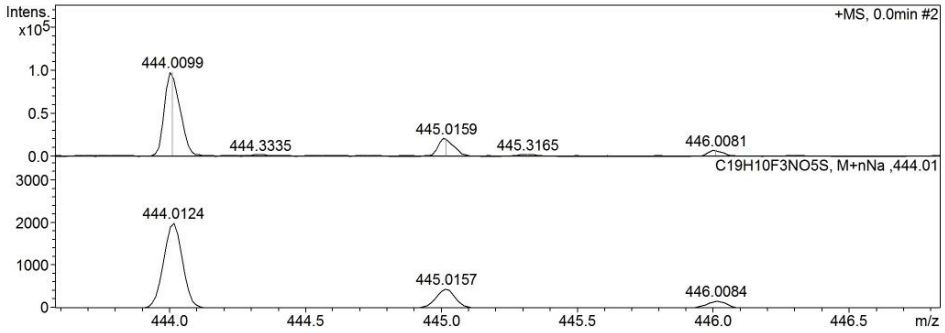
Acquisition Parameter

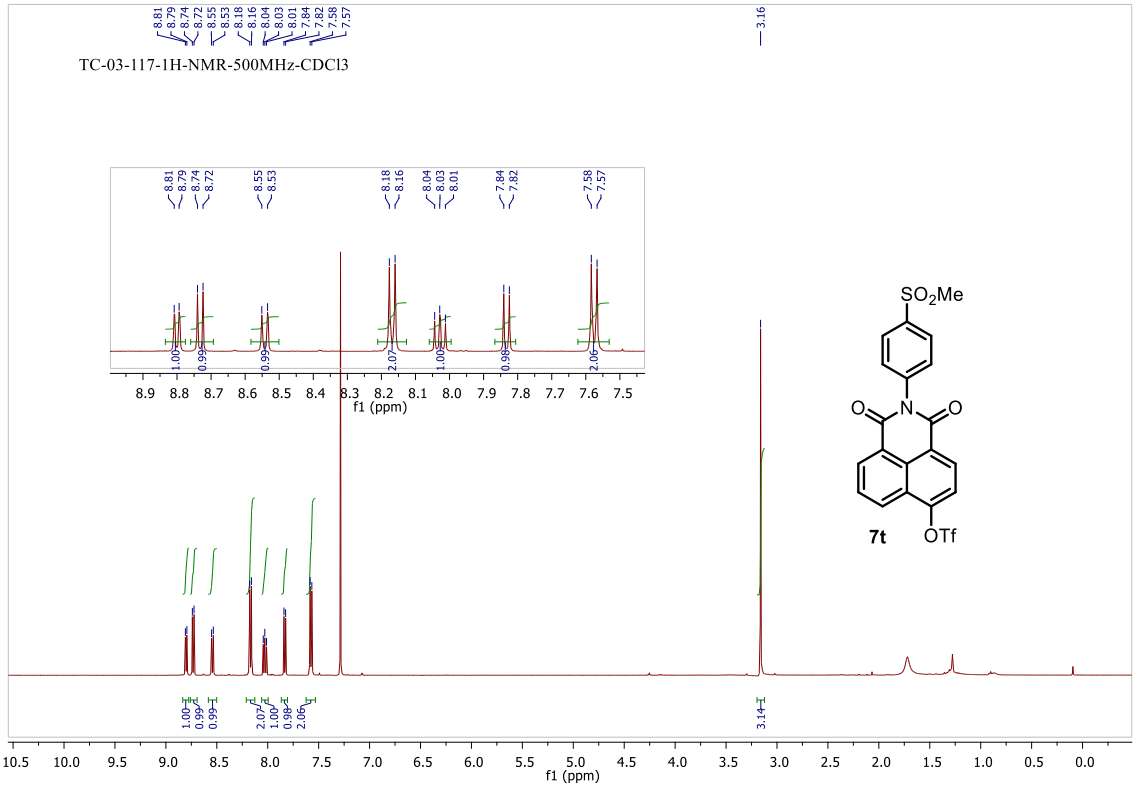
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.8 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

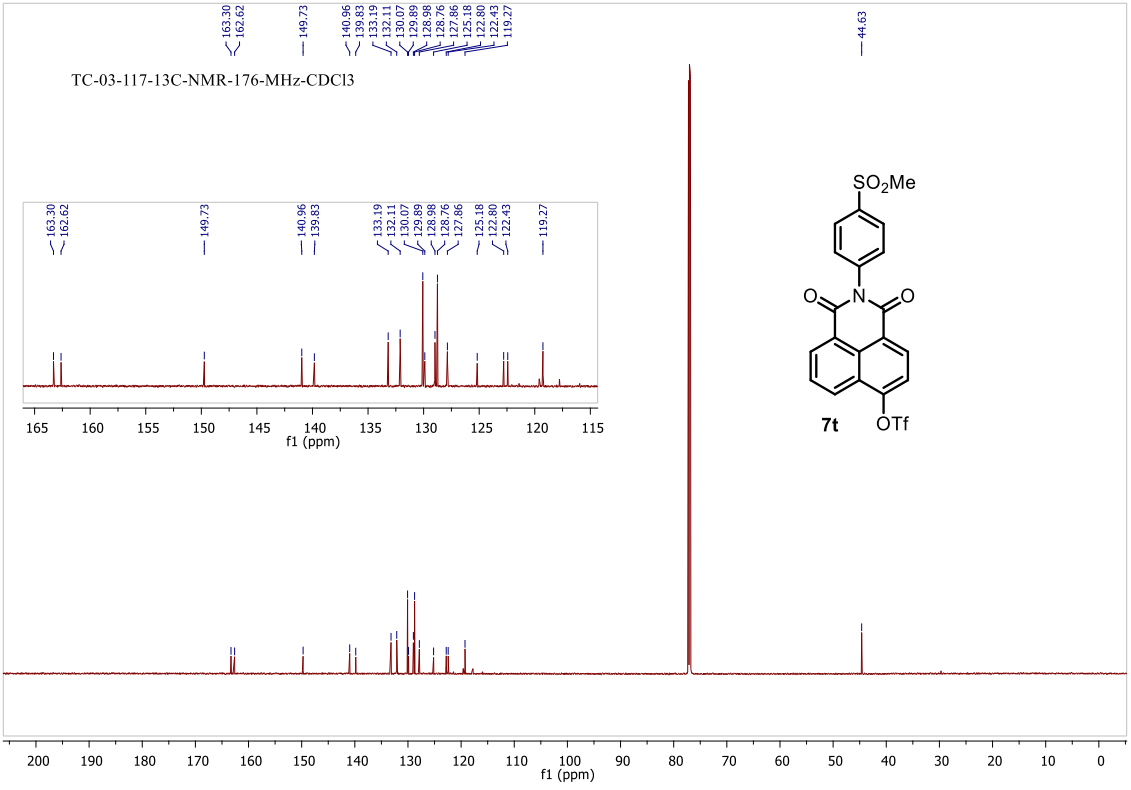


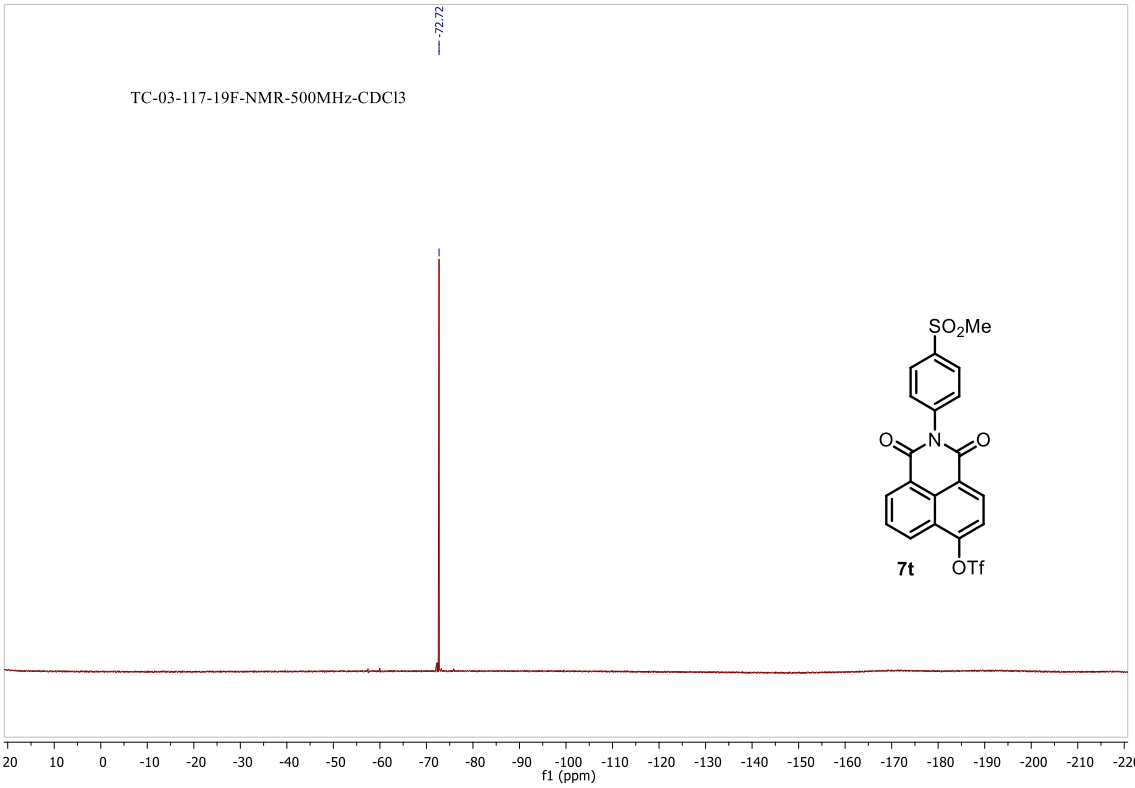
TIC +All MS






TC-03-117-1H-NMR-500MHz-CDCl3
8.81
8.79
8.74
8.72
8.55
8.53
8.18
8.16
8.04
8.03
8.01
7.84
7.82
7.58
7.57
3.16
SO2Me
O
N
O
7t
OTf
f1 (ppm)


TC-03-117-13C-NMR-176-MHz-CDCl3
163.30
162.62
149.73
140.96
139.83
133.19
132.11
130.07
129.89
128.98
128.76
127.86
125.18
122.80
122.43
119.27
44.63
SO2Me
O
N
O
7t
OTf
f1 (ppm)


-72.72
TC-03-117-19F-NMR-500MHz-CDCl3
SO2Me
O
N
O
7t
OTf
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

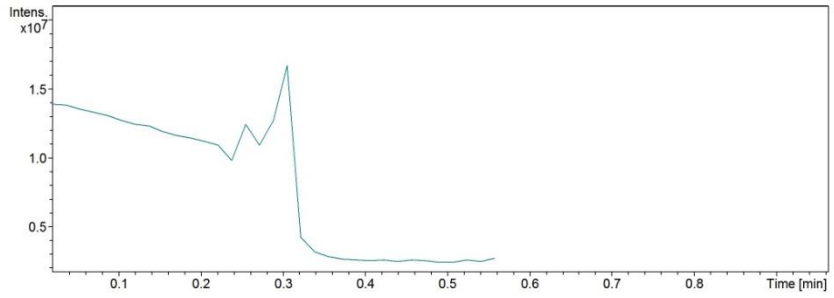
Display Report

Analysis Info

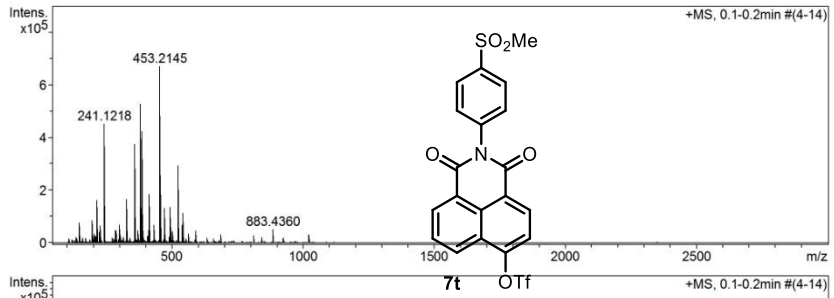
Analysis Name	D:\Data\USER DATA 2023\Nov23\20-nov\Dr M.Kapur-TC-03-117-1.d	Acquisition Date	20-11-2023 12:14:44
Method	tune_low_APR23.m	Operator	Bruker
Sample Name	-TC-03-117-1	Instrument	micrOTOF-Q 10330
Comment			

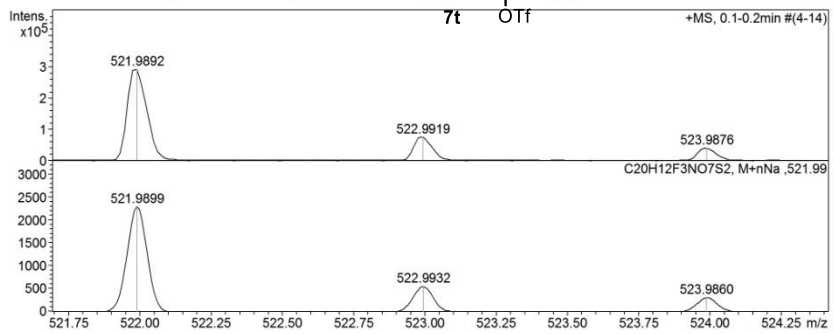
Acquisition Parameter

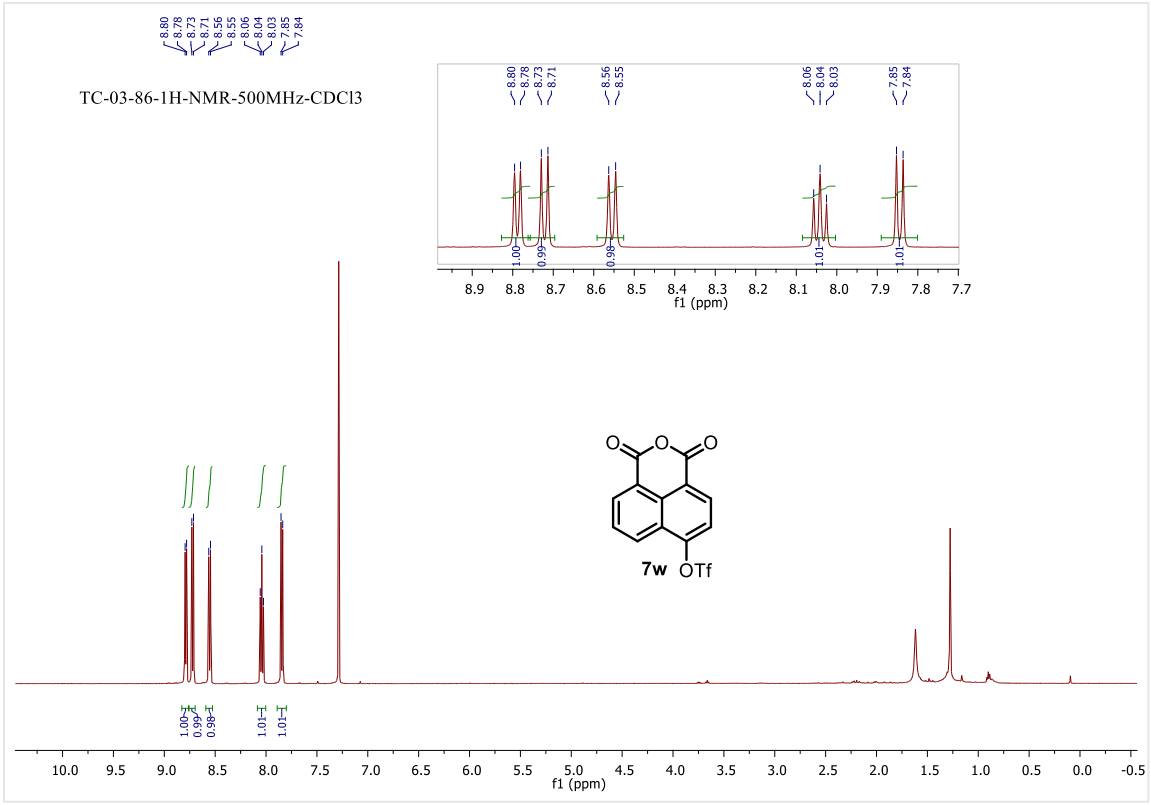
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

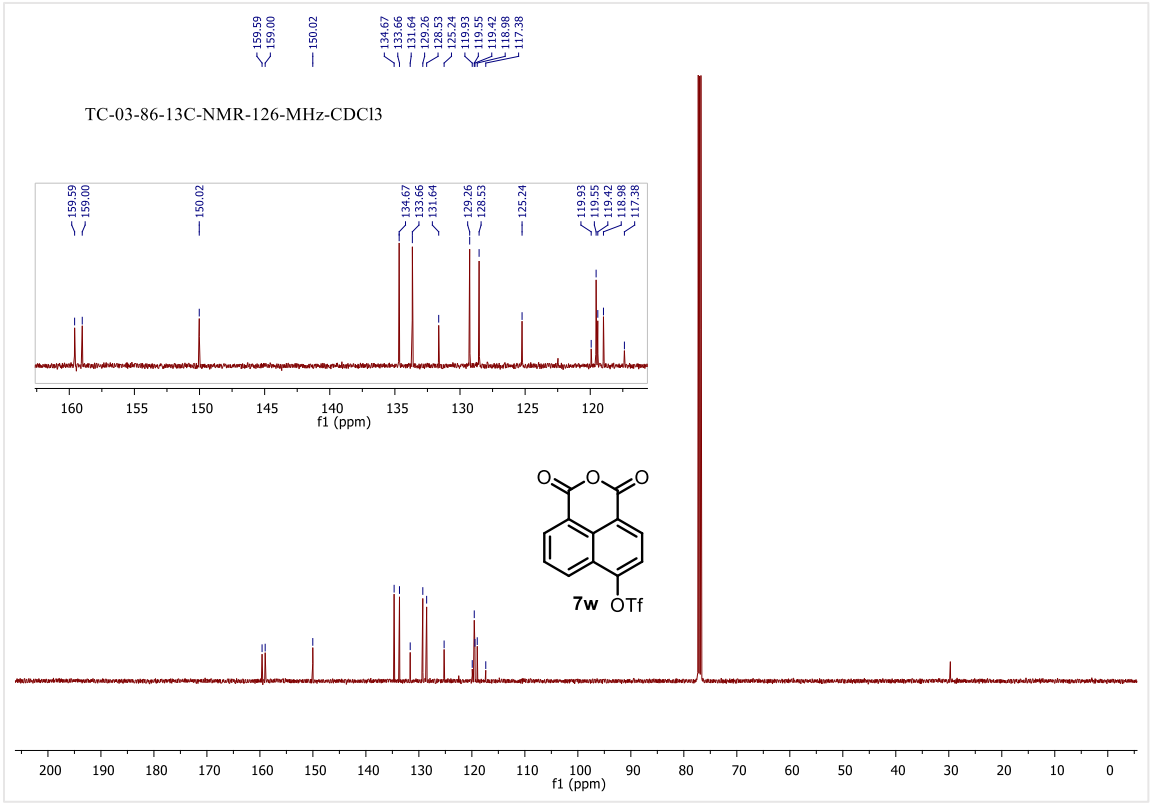


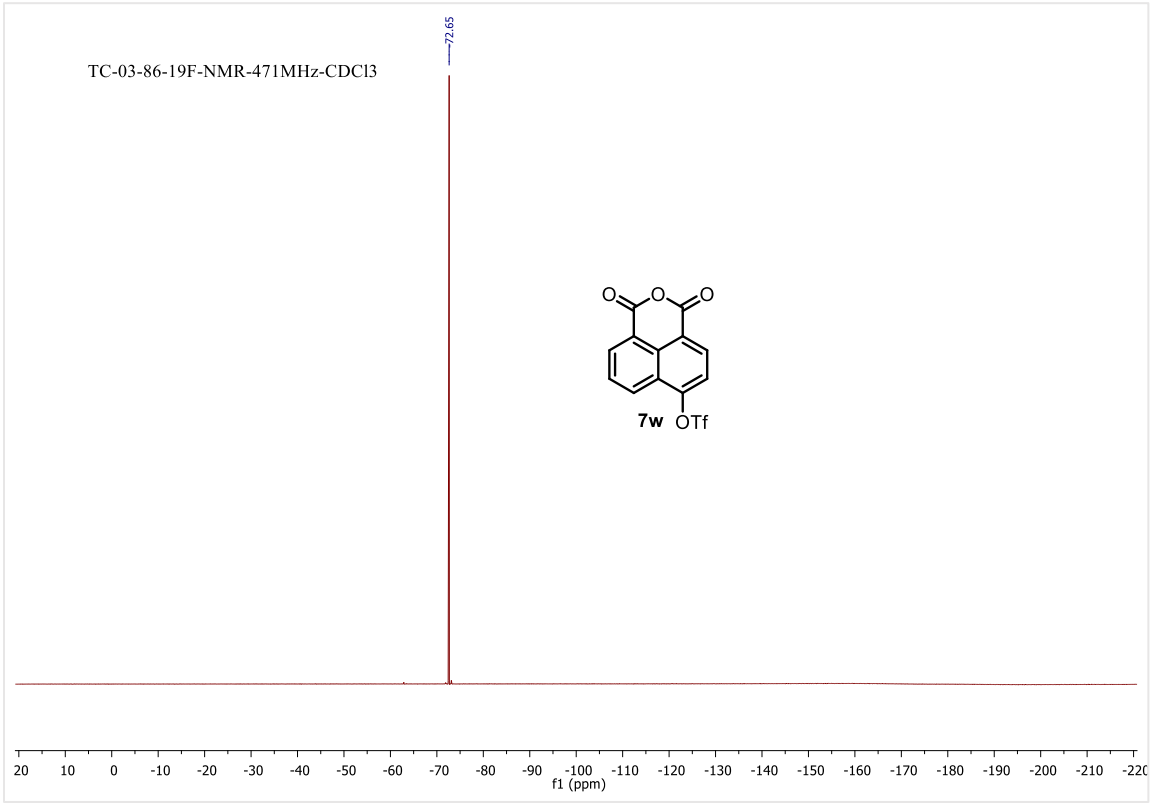
TIC +All MS






TC-03-86-1H-NMR-500MHz-CDCl3
8.80
8.78
8.73
8.71
8.56
8.55
8.06
8.04
8.03
7.85
7.84
1.00
0.99
0.98
1.01
1.01
f1 (ppm)
7w
OTf


159.59
159.00
150.02
134.67
133.66
131.64
129.26
128.53
125.24
119.93
119.55
119.42
118.98
117.38
TC-03-86-13C-NMR-126-MHz-CDCl3
f1 (ppm)
7w OTf
f1 (ppm)


72.65
TC-03-86-19F-NMR-471MHz-CDCl3
7w OTf
f1 (ppm)

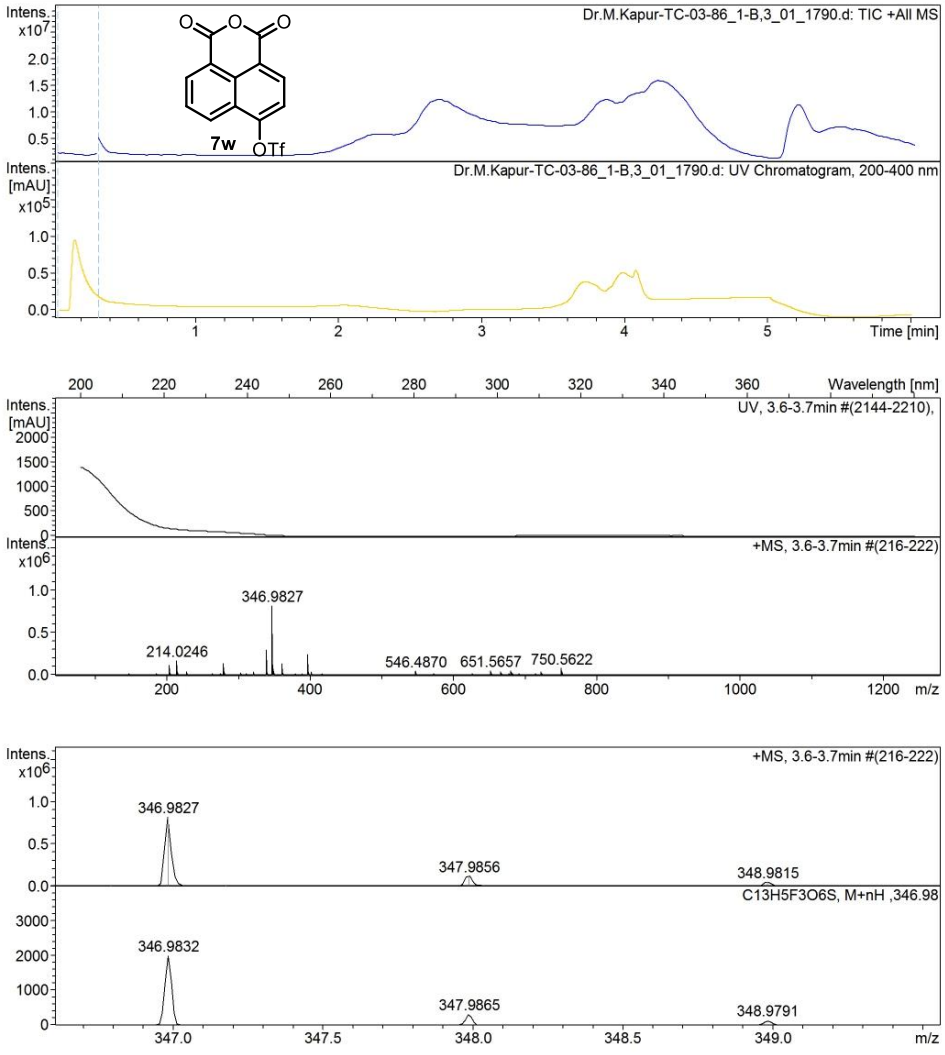
Display Report

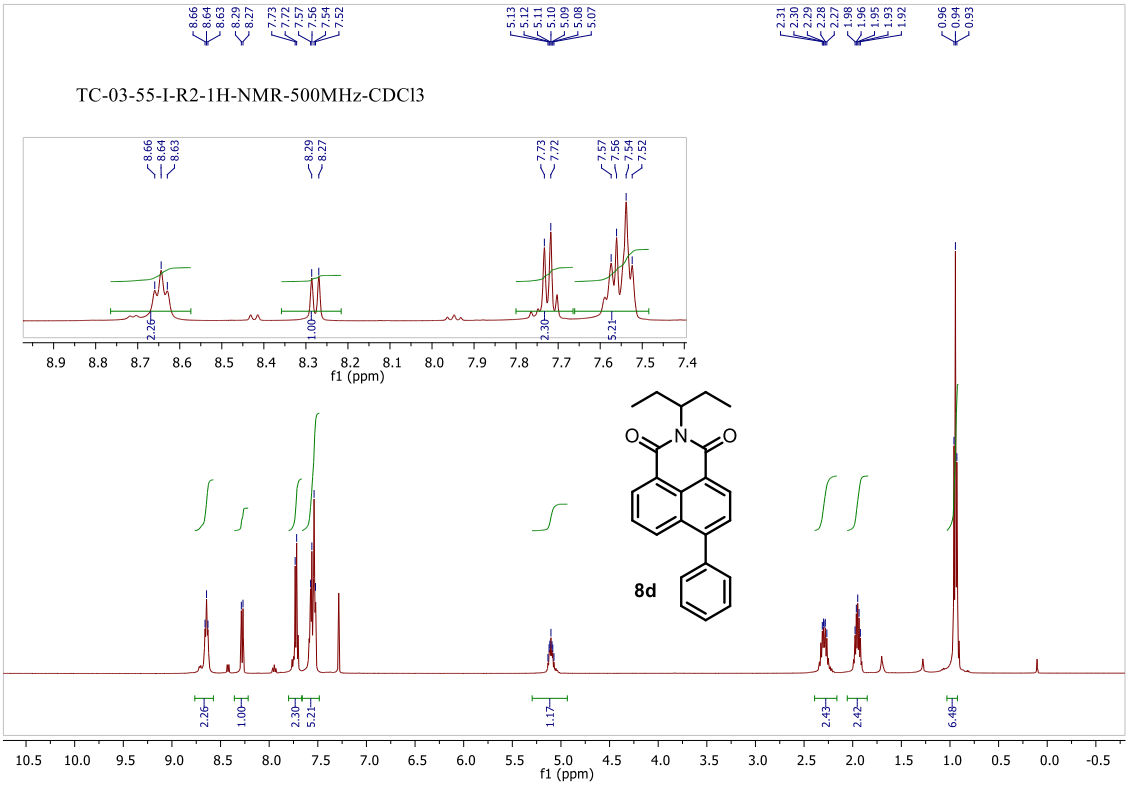
Analysis Info

Analysis Name	D:\Data\USER DATA 2023\June\09-june\Dr.M.Kapur-TC-03-86_1-B,3_01_1790.d	Acquisition Date	09-06-2023 13:12:25
Method	HRLCMS-20 APR23.m	Operator	Bruker
Sample Name	Dr.M.Kapur-TC-03-86	Instrument	micrOTOF-Q 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

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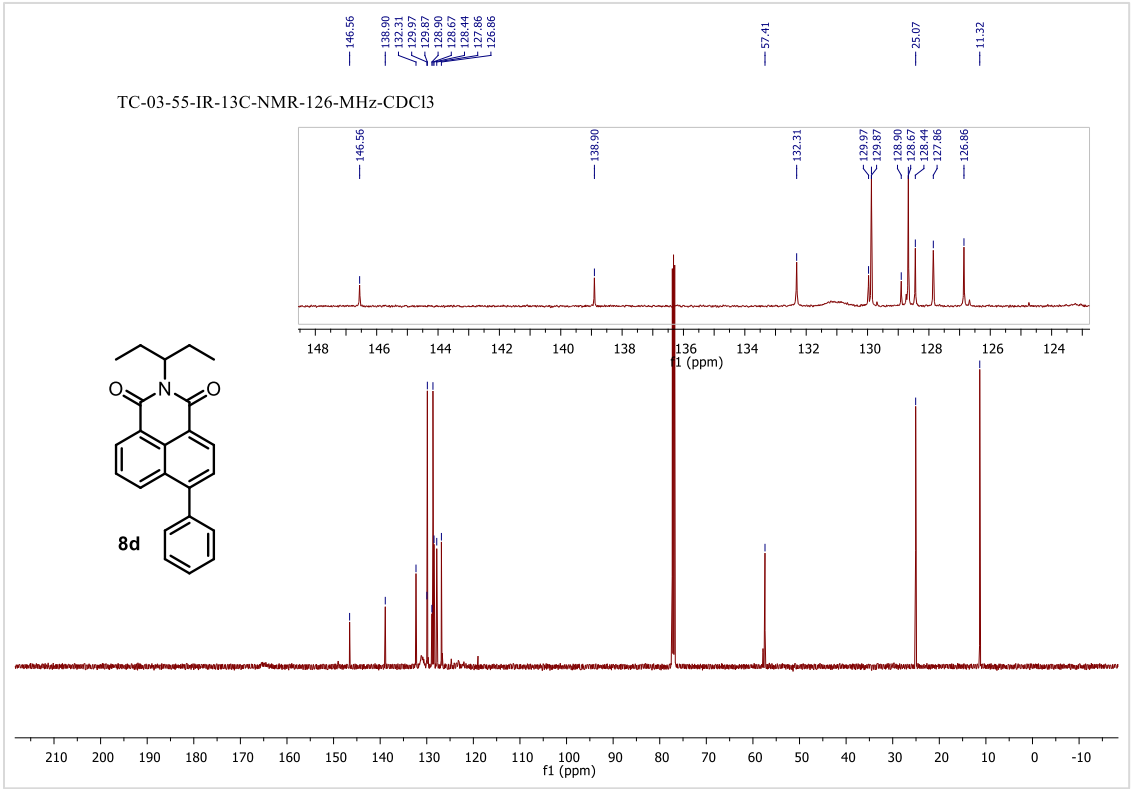
8d
f1 (ppm)


TC-03-55-IR-13C-NMR-126-MHz-CDCl3
8d
f1 (ppm)

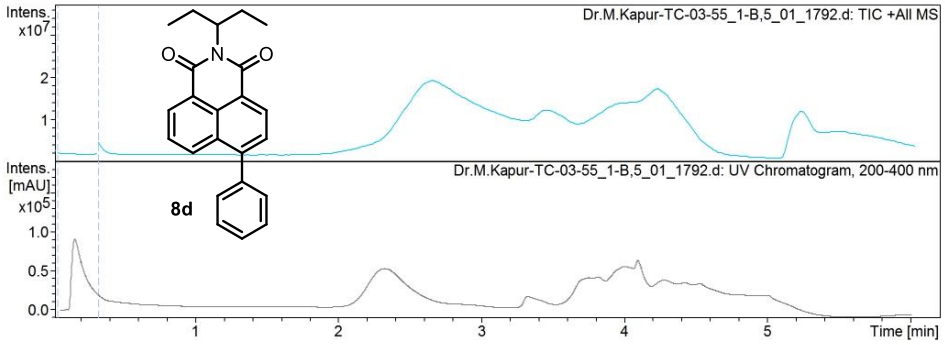
Display Report

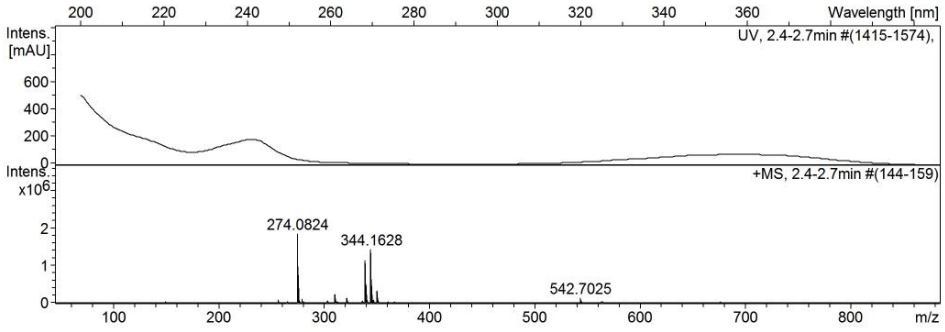
Analysis Info

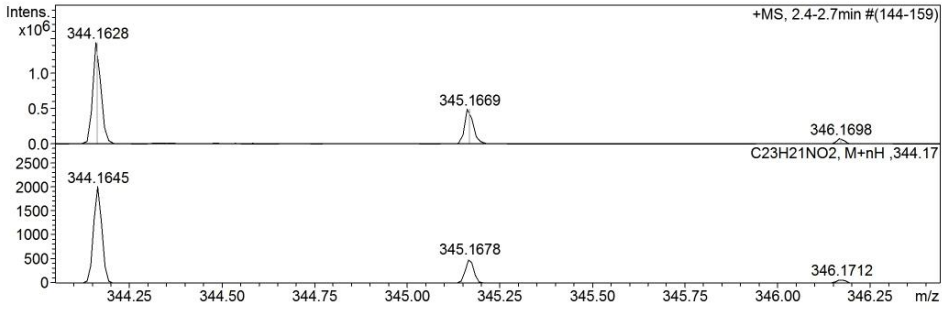
Analysis Name	D:\Data\USER DATA 2023\June\09-june\Dr.M.Kapur-TC-03-55_1-B,5_01_1792.d	Acquisition Date	09-06-2023 13:26:58
Method	HRLCMS-20 APR23.m	Operator	Bruker
Sample Name	Dr.M.Kapur-TC-03-55	Instrument	micrOTOF-Q 10330
Comment			

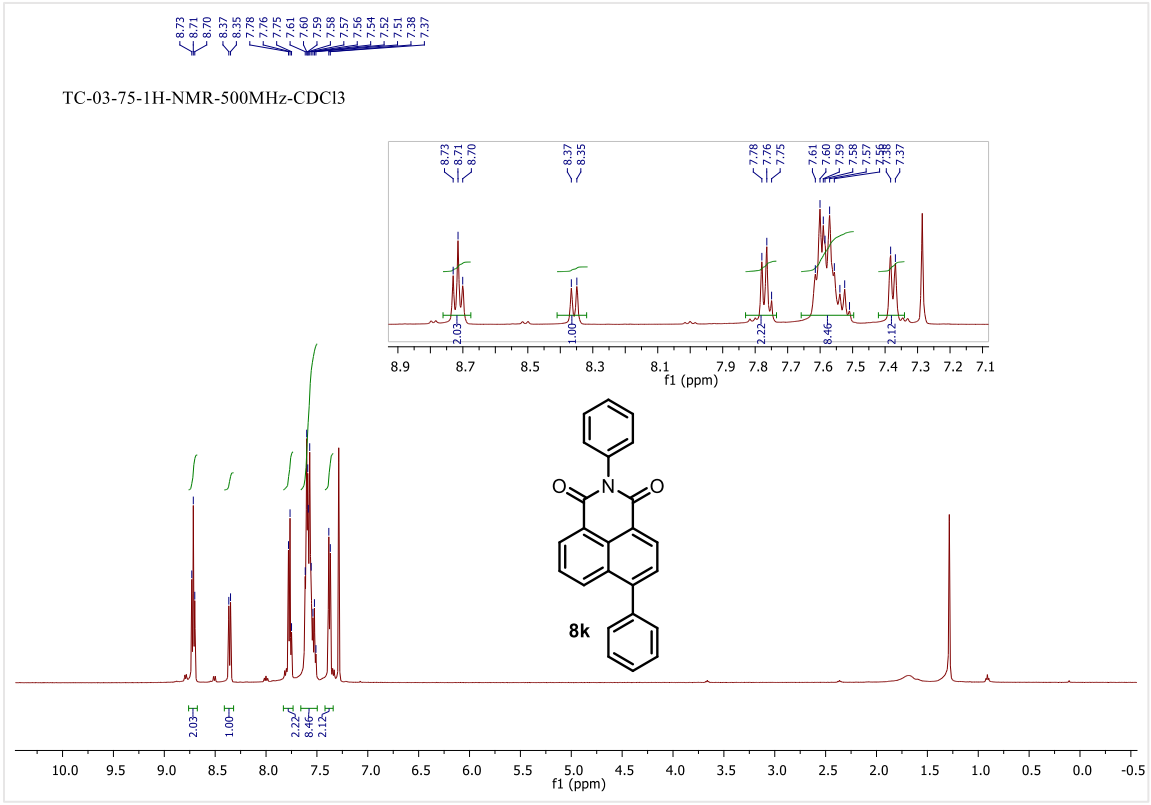
Acquisition Parameter

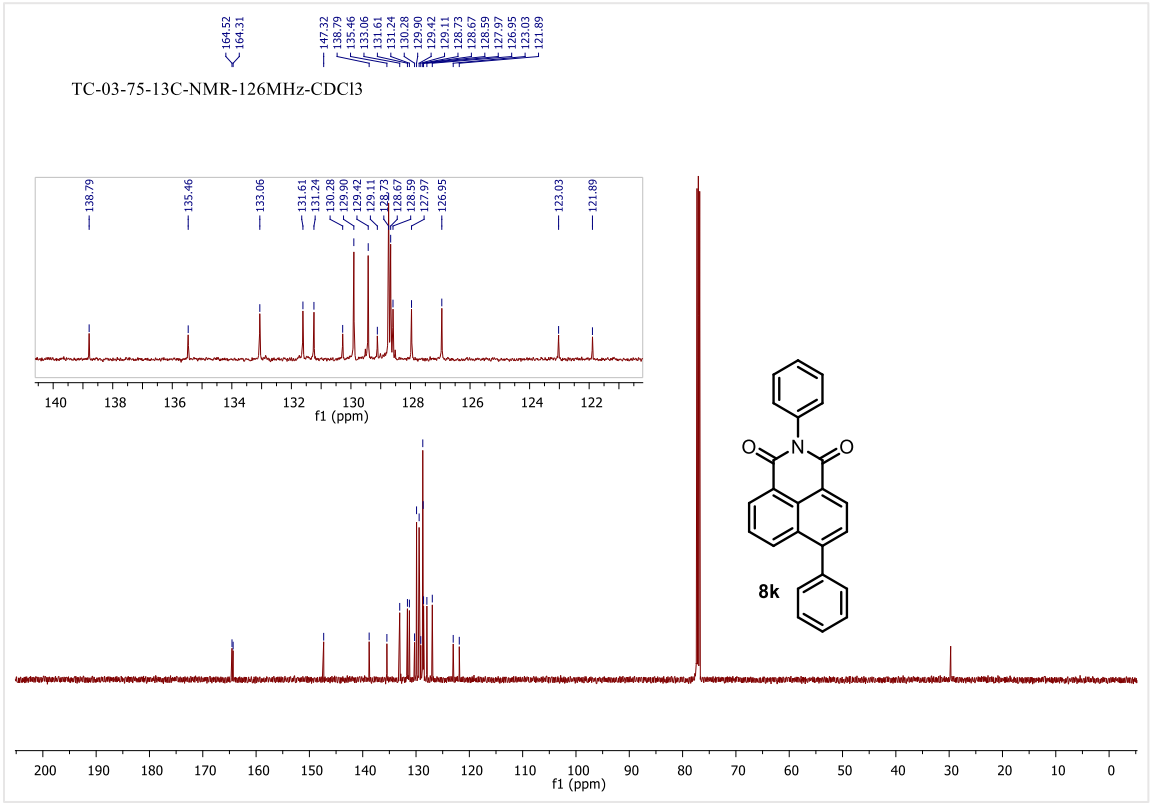
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste








TC-03-75-1H-NMR-500MHz-CDCl3
8k
f1 (ppm)


TC-03-75-13C-NMR-126MHz-CDCl3
8k
f1 (ppm)

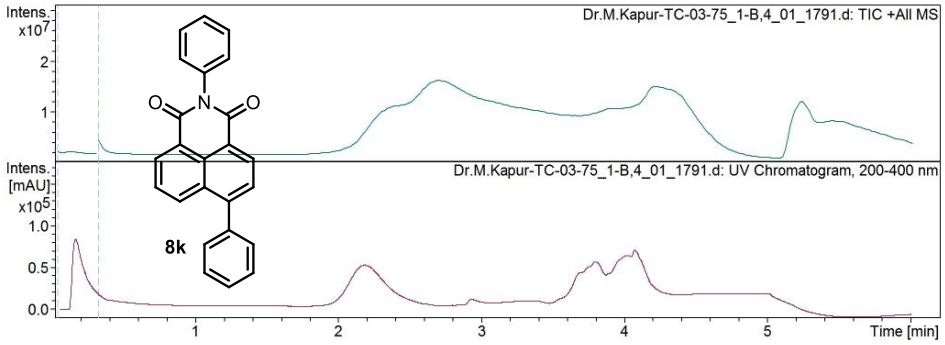
Display Report

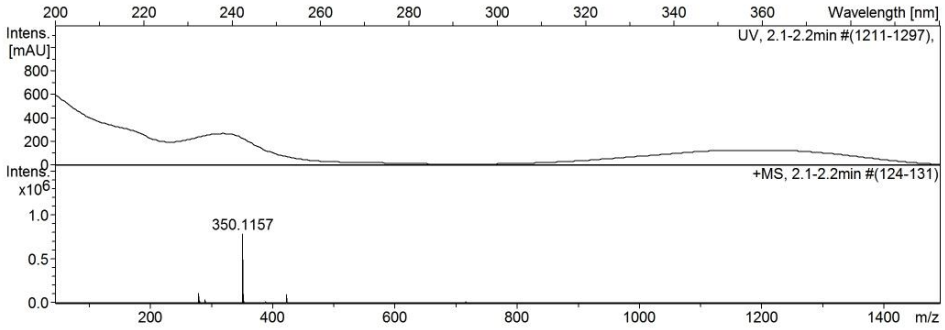
Analysis Info

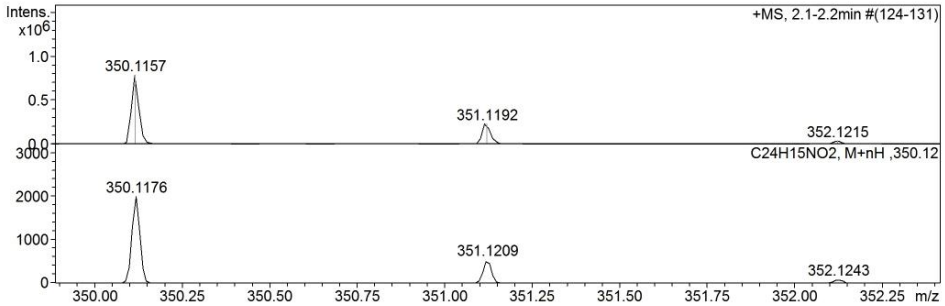
Analysis Name	D:\Data\USER DATA 2023\June\09-june\Dr.M.Kapur-TC-03-75_1-B,4_01_1791.d	Acquisition Date	09-06-2023 13:19:41
Method	HRLCMS-20 APR23.m	Operator	Bruker
Sample Name	Dr.M.Kapur-TC-03-75	Instrument	micrOTOF-Q 10330
Comment			

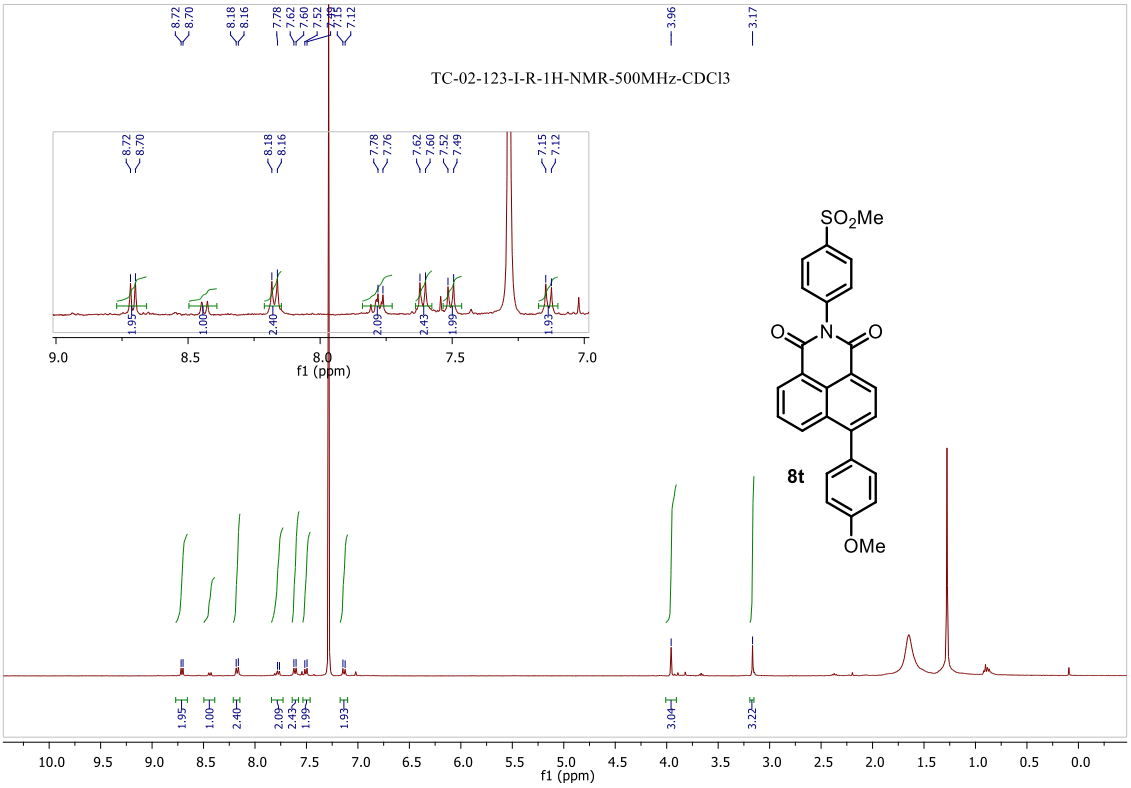
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste








TC-02-123-I-R-1H-NMR-500MHz-CDCl3
8.72
8.70
8.18
8.16
7.78
7.62
7.60
7.52
7.49
7.15
7.12
3.96
3.17
f1 (ppm)
SO2Me
O
N
O
8t
OMe

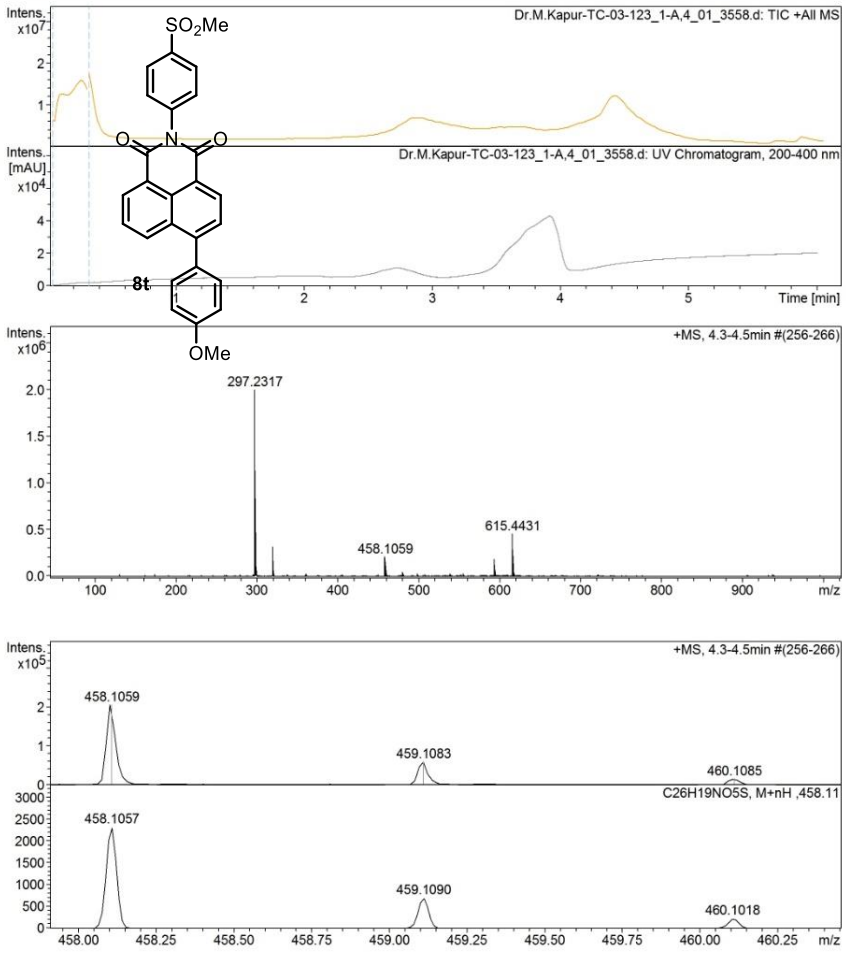
Display Report

Analysis Info

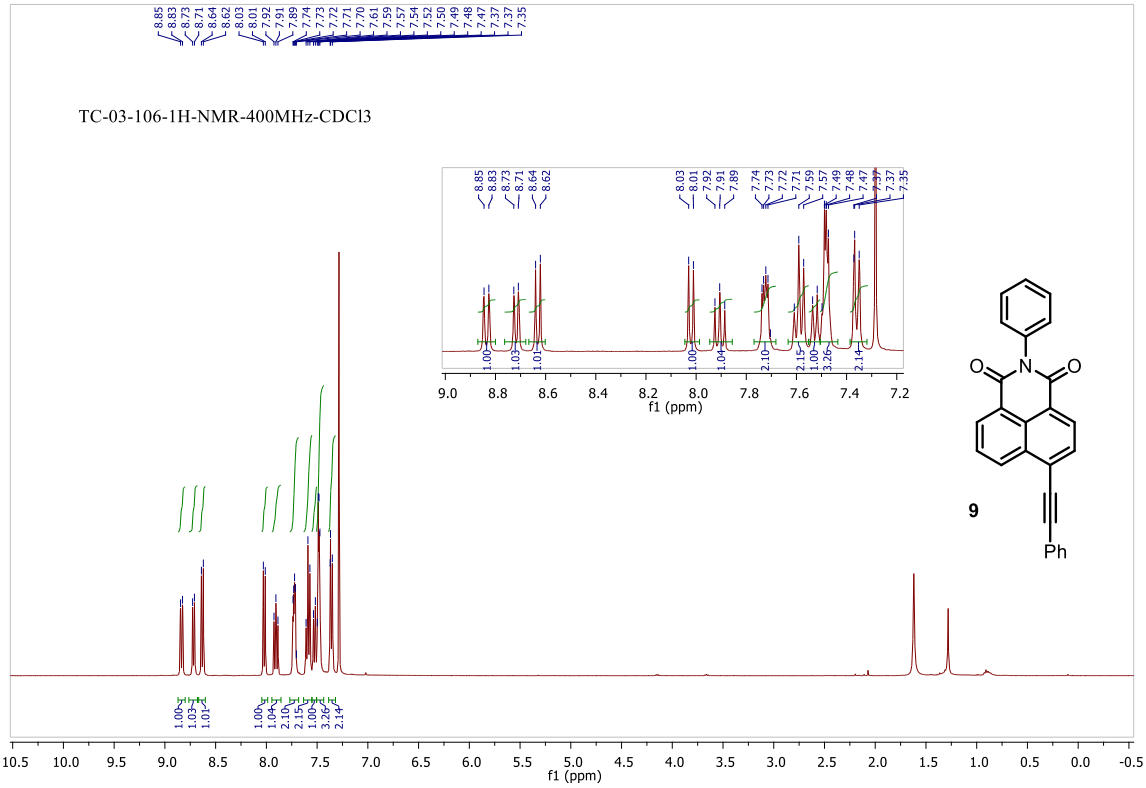
Analysis Name	D:\Data\USER DATA 2023\Dec23\01-Dec\Dr.M.Kapur-TC-03-123_1-A,4_01_3558.d	Acquisition Date	01-12-2023 11:33:40
Method	HRLCMS-20 Sept.m	Operator	Bruker
Sample Name	Dr.M.Kapur-TC-03-123	Instrument	micrOTOF-Q 10330
Comment			

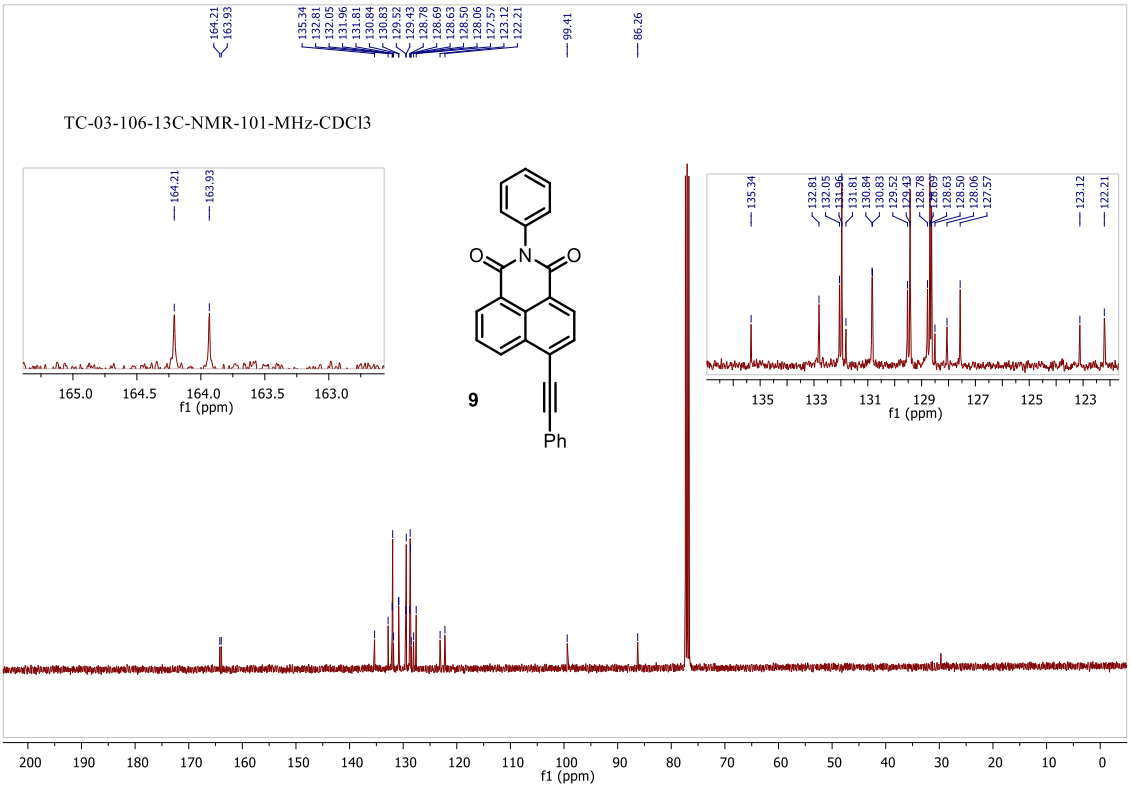
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0 printed: 01-12-2023 14:47:11 Page 1 of 1


TC-03-106-1H-NMR-400MHz-CDCl3
9
Ph
f1 (ppm)


TC-03-106-13C-NMR-101-MHz-CDCl3
9
Ph
f1 (ppm)

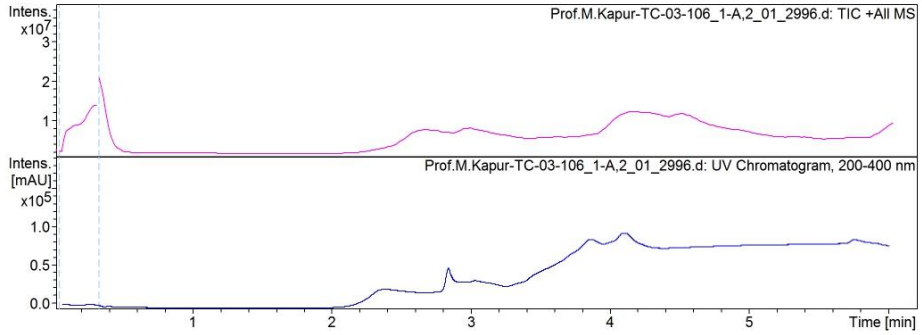
Display Report

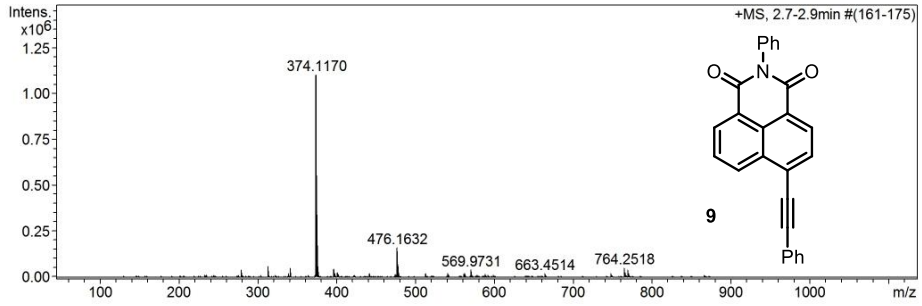
Analysis Info

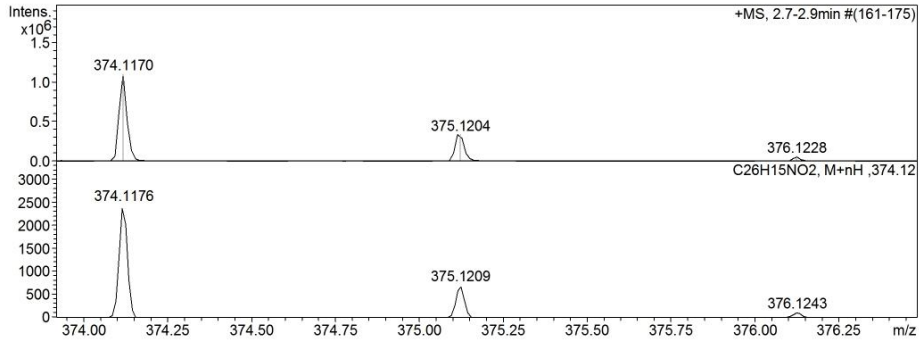
Analysis Name	D:\Data\USER DATA 2023\sep 23\12-SEP\Prof.M.Kapur-TC-03-106_1-A,2_01_2996.d	Acquisition Date	12-09-2023 10:20:38
Method	HRLCMS-20 APR23.m	Operator	Bruker
Sample Name	Prof.M.Kapur-TC-03-106	Instrument	micrOTOF-Q 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste







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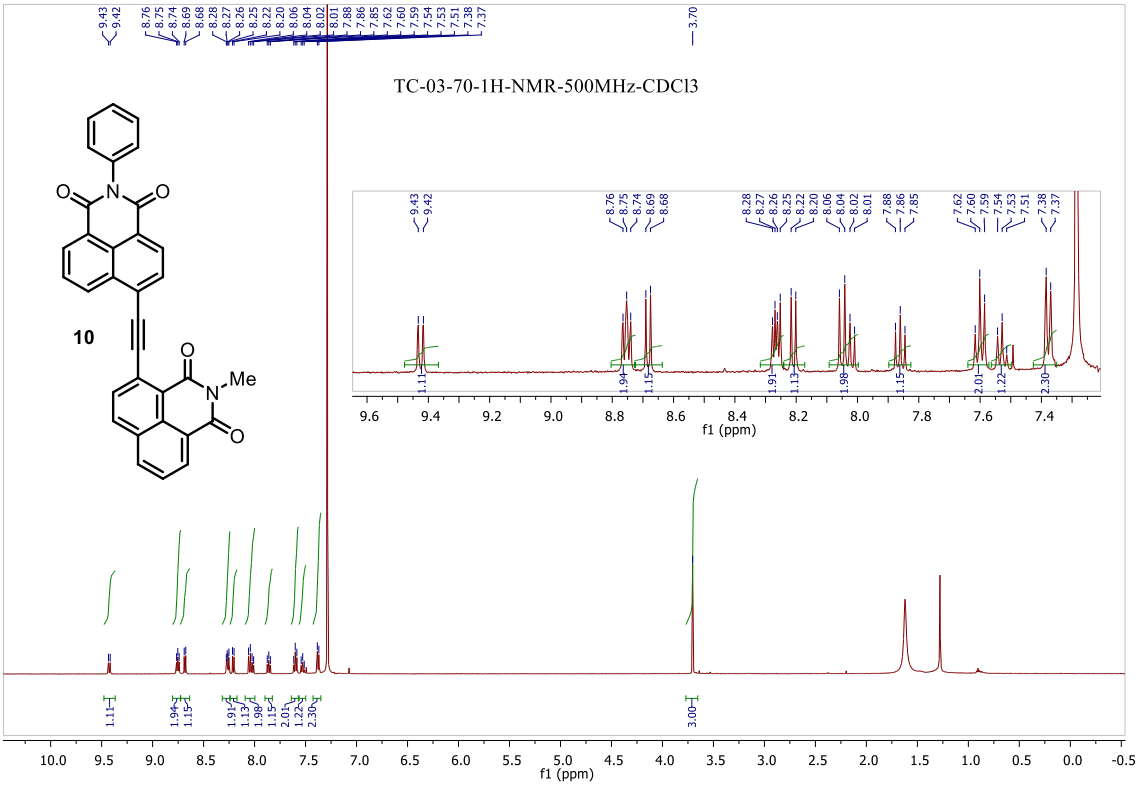
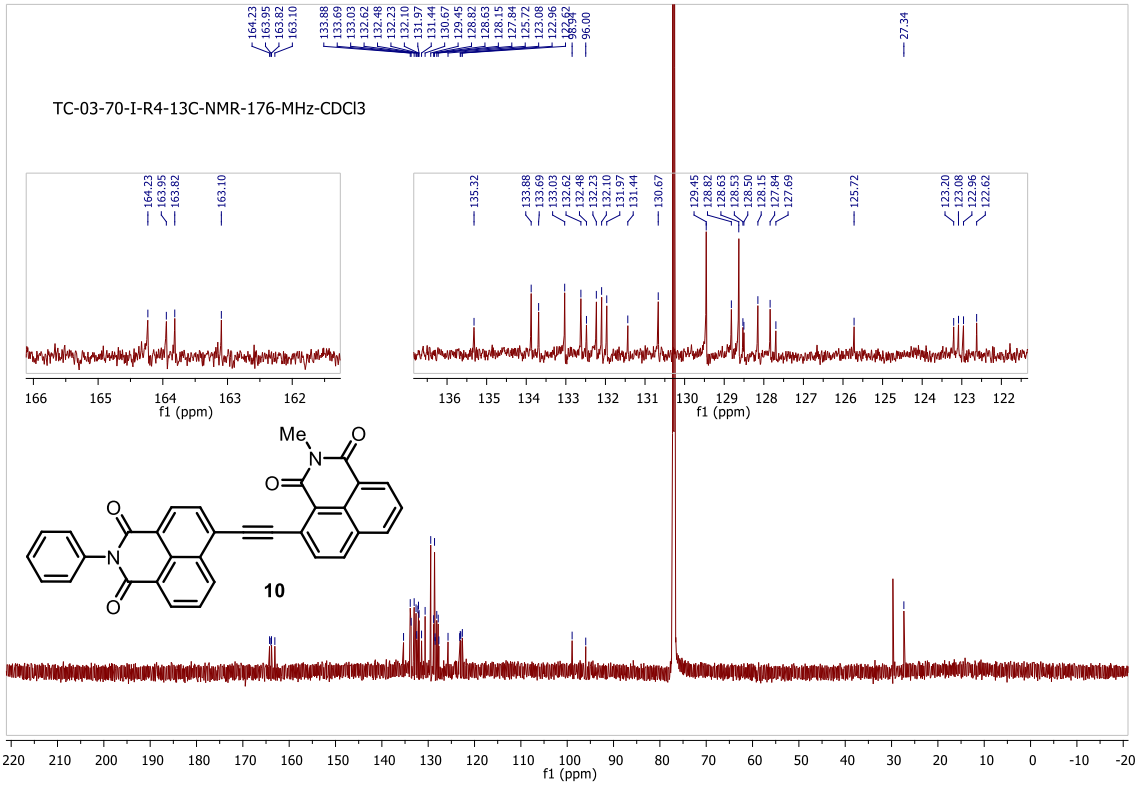

TC-03-70-1H-NMR-500MHz-CDCl3
10
f1 (ppm)


TC-03-70-I-R4-13C-NMR-176-MHz-CDCl3
10
f1 (ppm)

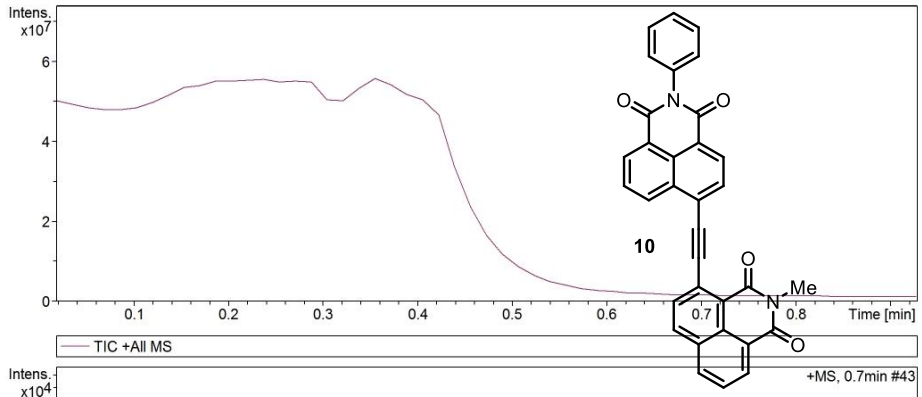
Display Report

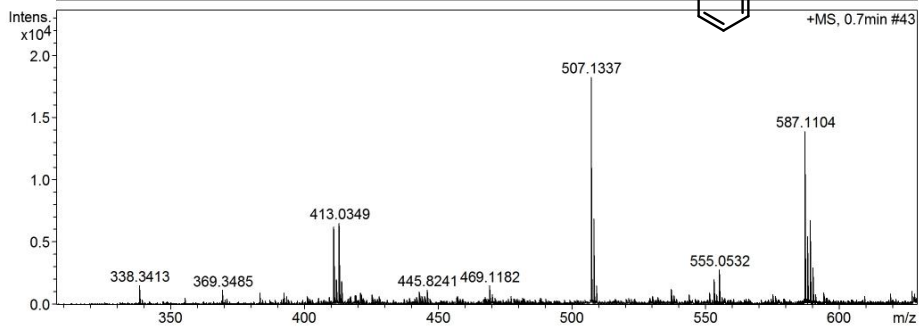
Analysis Info

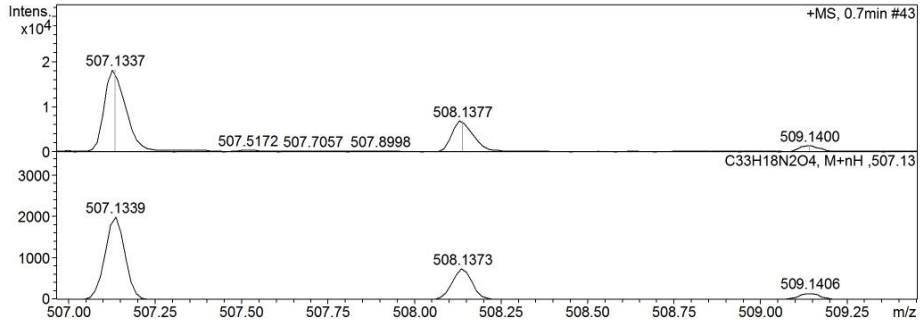
Analysis Name	D:\Data\Data-USER DATA 2023-Mar-03-03-23\Dr.M.Kapur-TC-03-70-1.d	Acquisition Date	03-03-2023 15:37:31
Method	tune_wide_Jan_2019.m	Operator	Bruker
Sample Name	-TC-03-70-1	Instrument	micrOTOF-Q 10330
Comment			

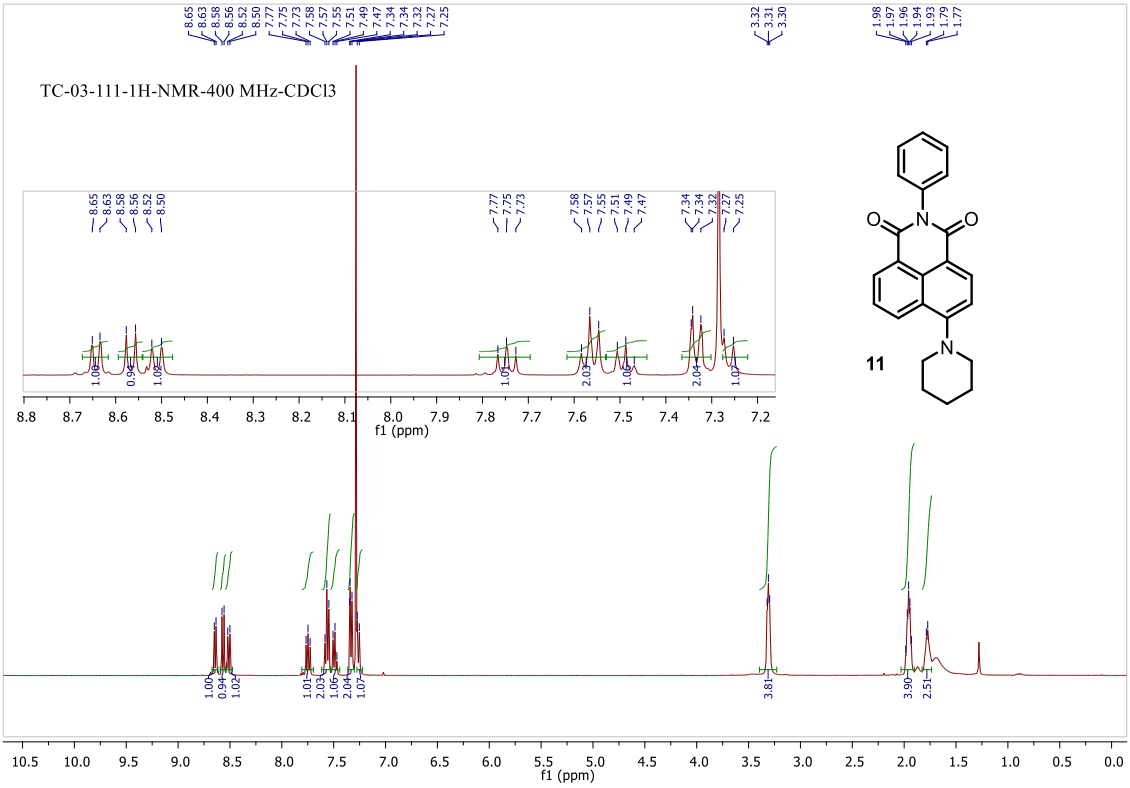
Acquisition Parameter

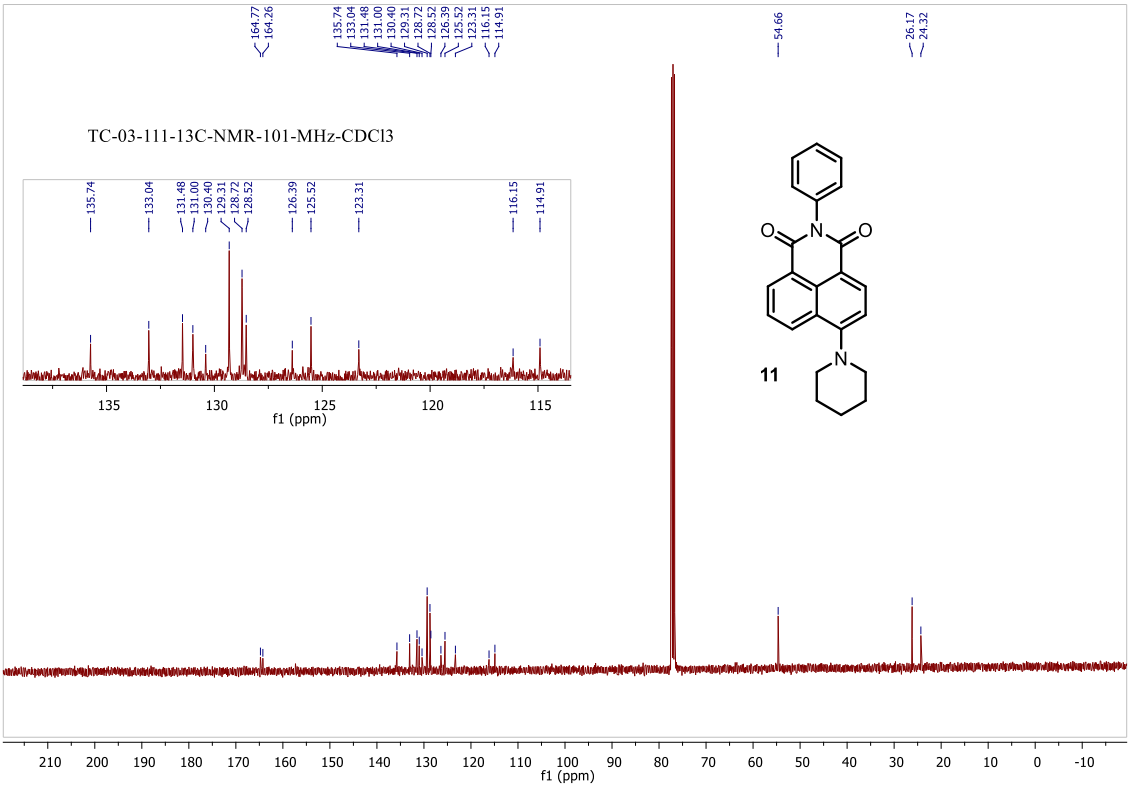
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	5.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste








TC-03-111-1H-NMR-400 MHz-CDCl3
11
f1 (ppm)


TC-03-111-13C-NMR-101-MHz-CDCl3
11
f1 (ppm)

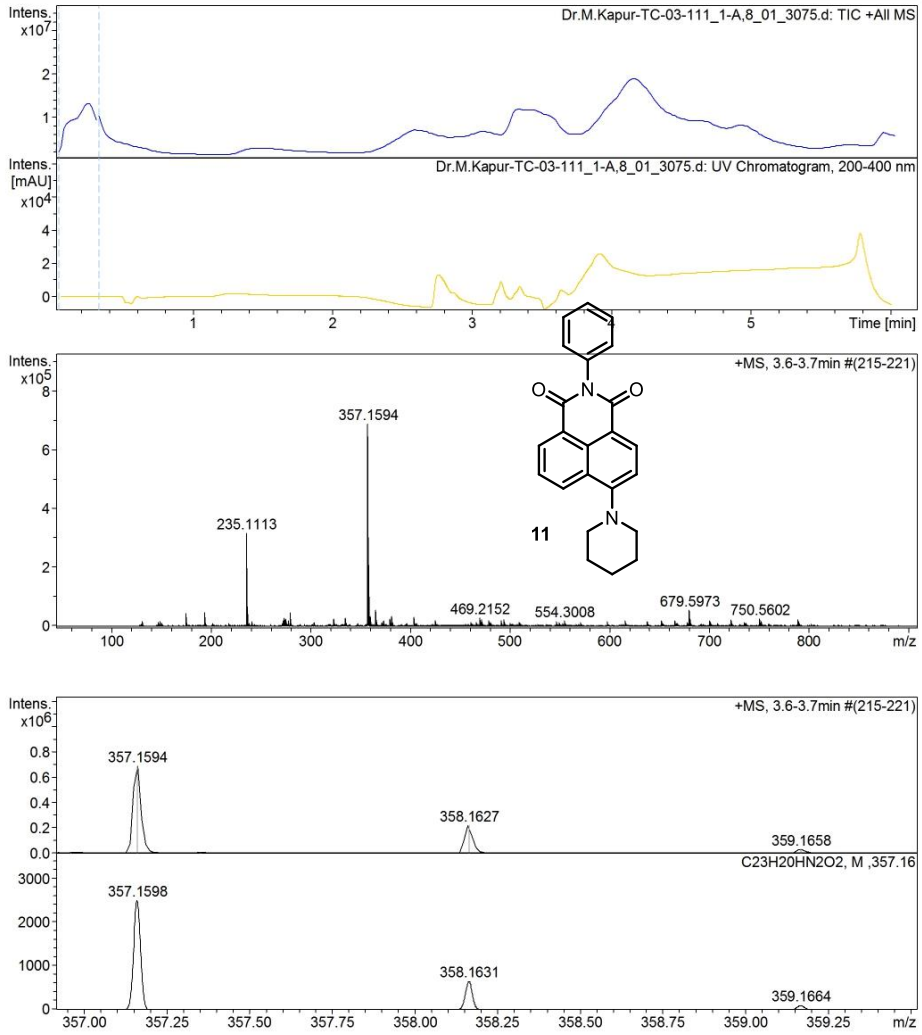
Display Report

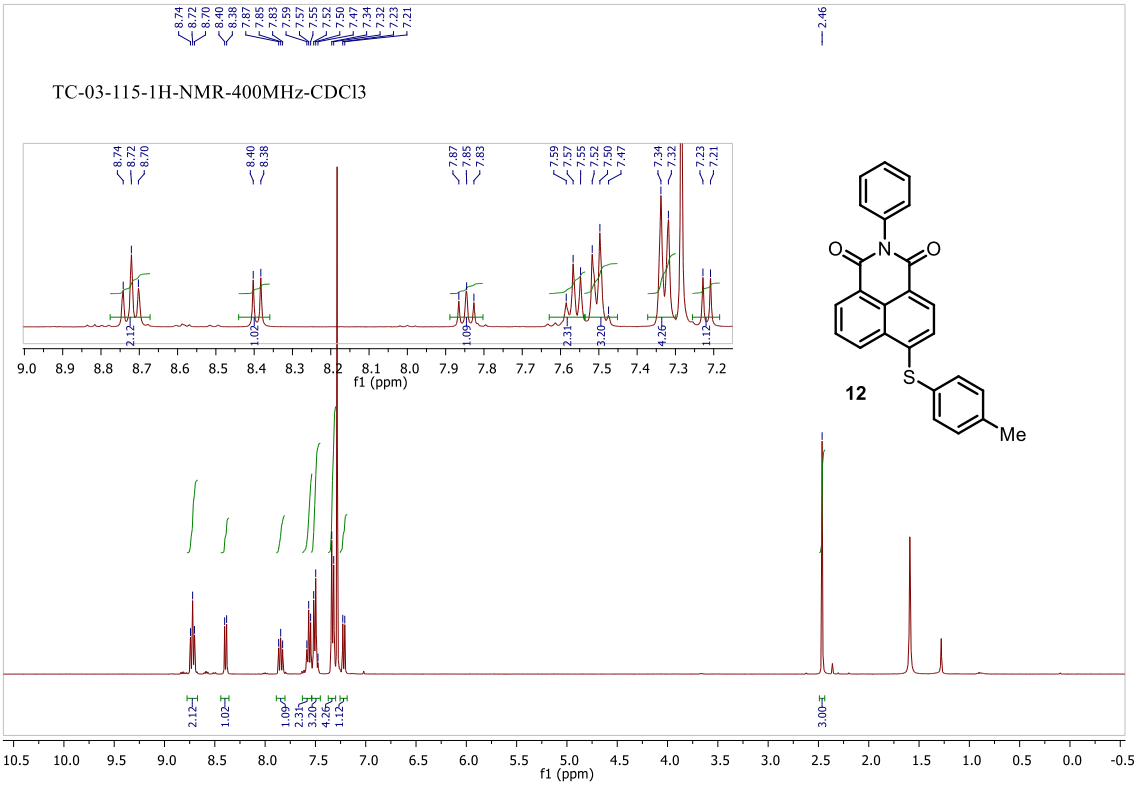
Analysis Info

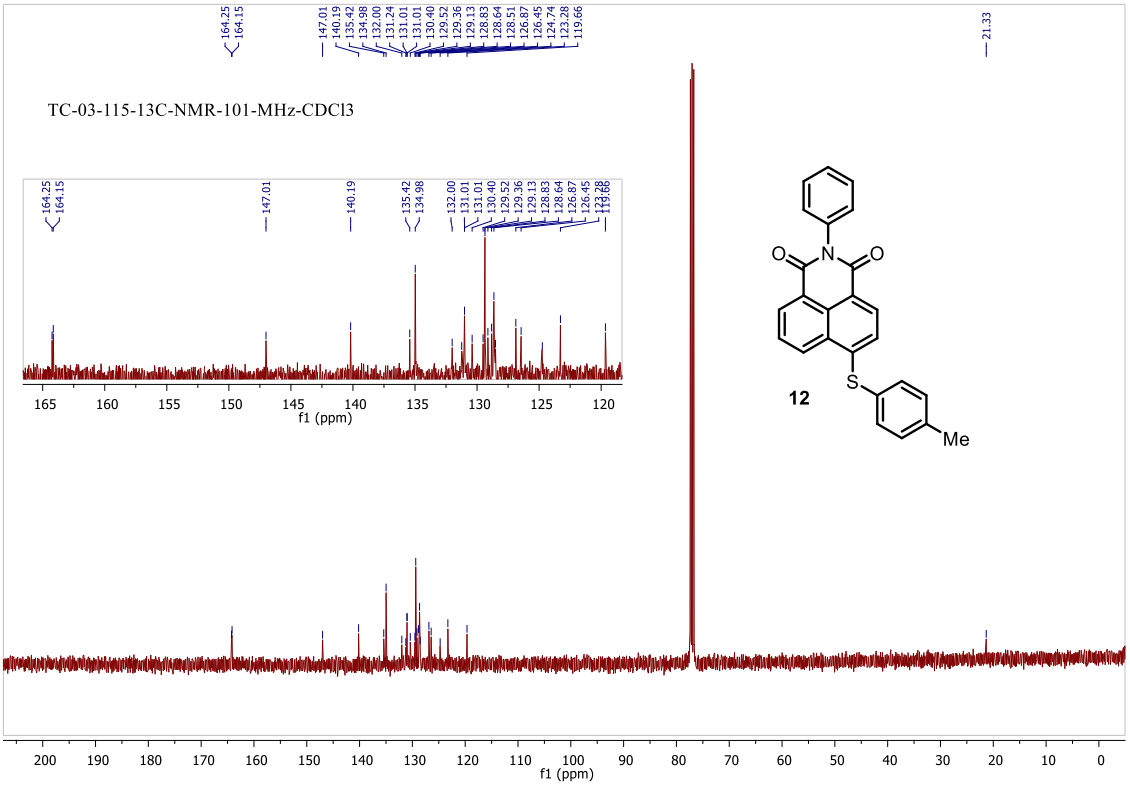
Analysis Name	D:\Data\USER DATA 2023\sep 23\22-sep\Dr.M.Kapur-TC-03-111_1-A,8_01_3075.d	Acquisition Date	22-09-2023 13:06:58
Method	HRLCMS-20 APR23.m	Operator	Bruker
Sample Name	Dr.M.Kapur-TC-03-111	Instrument	micrOTOF-Q 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste




TC-03-115-1H-NMR-400MHz-CDCl3
12
Me
f1 (ppm)


TC-03-115-13C-NMR-101-MHz-CDCl3
12
Me
f1 (ppm)

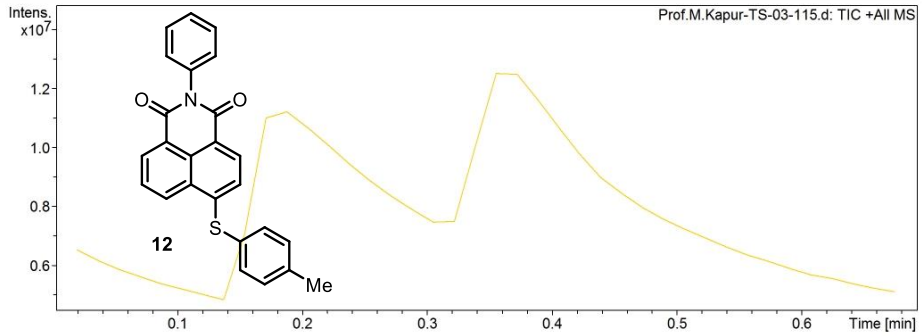
Display Report

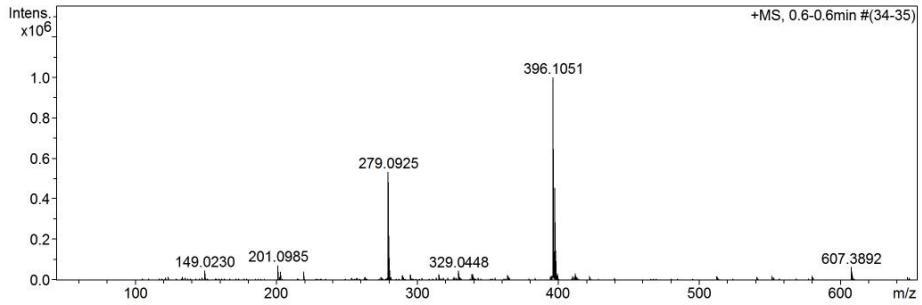
Analysis Info

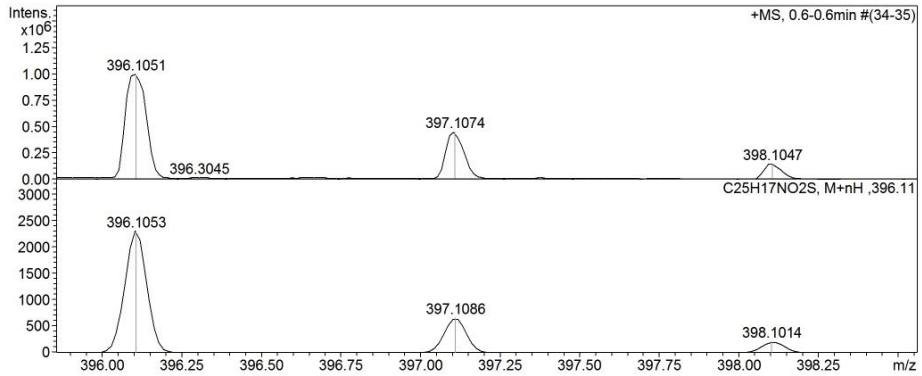
Analysis Name	D:\Data\USER DATA 2023\Oct 23\19-oct\Prof.M.Kapur-TS-03-115.d	Acquisition Date	19-10-2023 14:42:52
Method	tune_low_APCI.m	Operator	Bruker
Sample Name	TS-03-115	Instrument	micrOTOF-Q 10330
Comment			

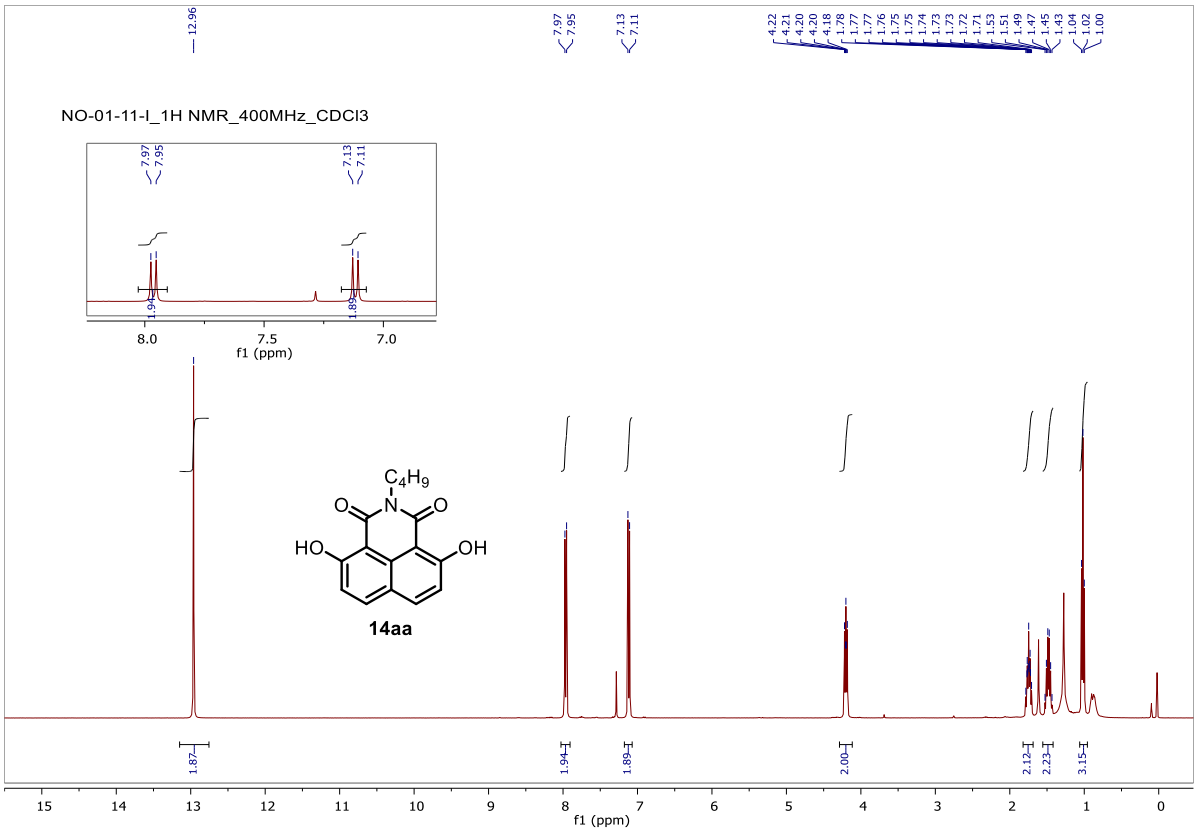
Acquisition Parameter

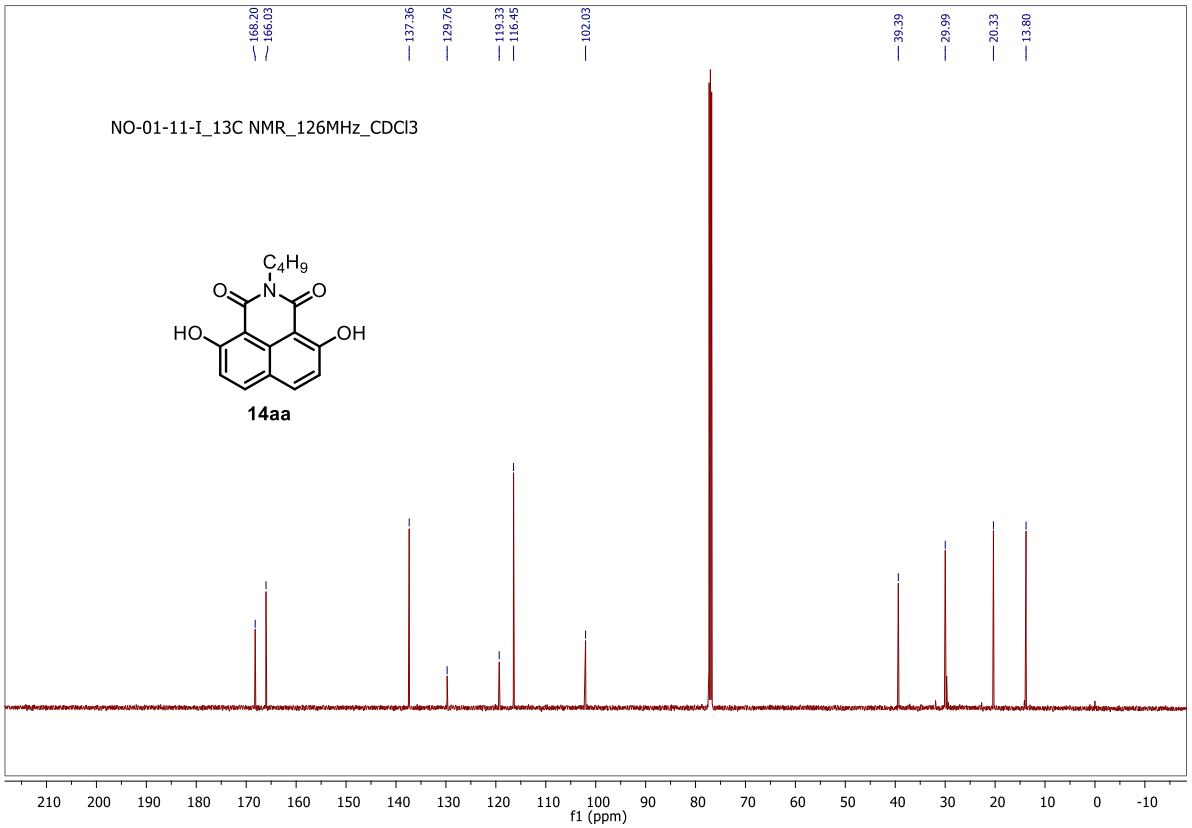
Source Type	Multi Mode	Ion Polarity	Positive	Set Nebulizer	2.0 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	5.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste








NO-01-11-I_1H NMR_400MHz_CDCl3
12.96
7.97
7.95
7.13
7.11
4.22
4.21
4.20
4.20
4.18
1.78
1.77
1.77
1.76
1.75
1.75
1.74
1.73
1.73
1.72
1.71
1.53
1.51
1.49
1.47
1.45
1.43
1.04
1.02
1.00
1.87
1.94
1.89
2.00
2.12
2.23
3.15
f1 (ppm)
14aa


NO-01-11-I_13C NMR_126MHz_CDCl3
168.20
166.03
137.36
129.76
119.33
116.45
102.03
39.39
29.99
20.33
13.80
f1 (ppm)
14aa

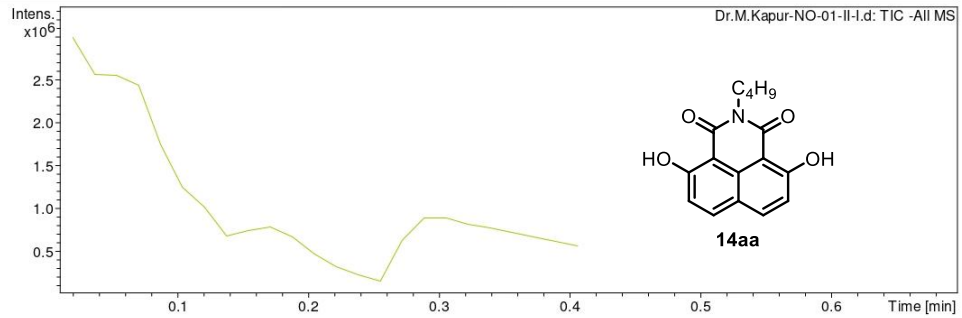
Display Report

Analysis Info

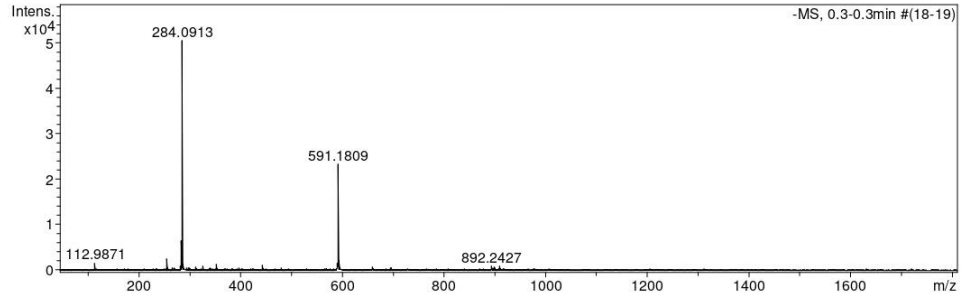
Analysis Name	D:\Data\new user data 2021\Dec-2021\1-Dec\Dr.M.Kapur-NO-01-II-I.d	Acquisition Date	12/1/2021 11:50:58 AM
Method	tune_low_neg.m	Operator	RUCHI
Sample Name	NO-01-II-I	Instrument	micrOTOF-Q II 10330
Comment			

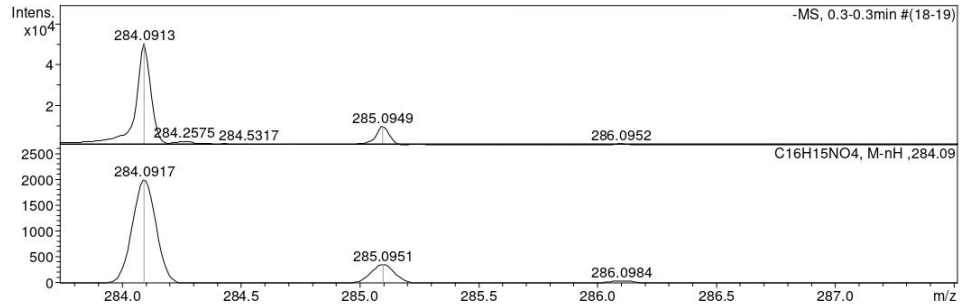
Acquisition Parameter

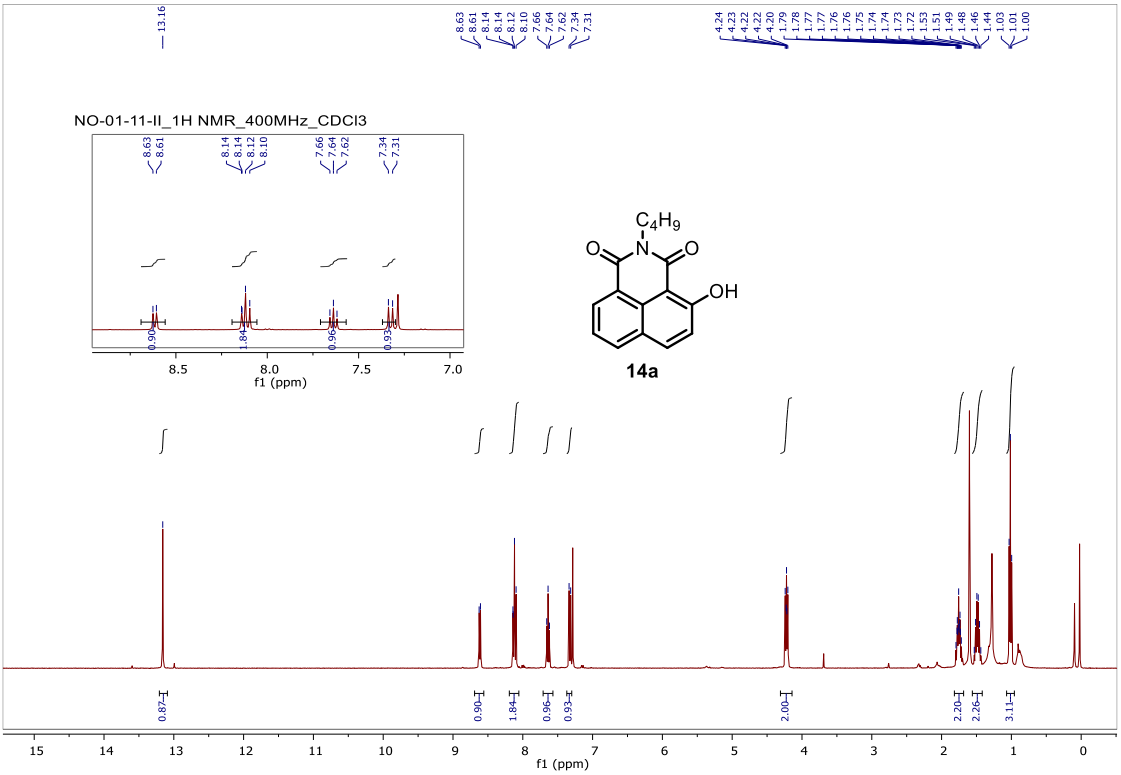
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

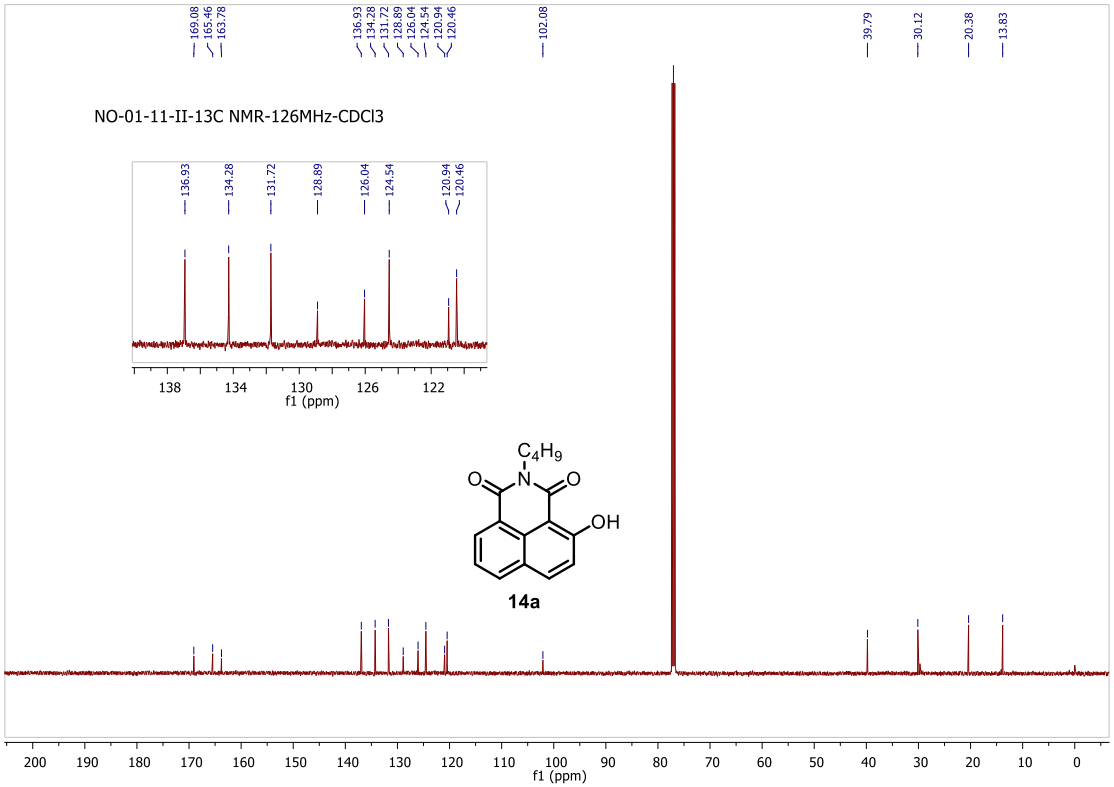


14aa






NO-01-11-II_1H NMR_400MHz_CDCl3
13.16
8.63
8.61
8.14
8.14
8.12
8.10
7.66
7.64
7.62
7.34
7.31
4.24
4.23
4.22
4.22
4.20
1.79
1.78
1.77
1.77
1.76
1.75
1.74
1.74
1.73
1.72
1.53
1.51
1.49
1.48
1.46
1.44
1.03
1.01
1.00
0.87
0.90
1.84
0.96
0.93
2.00
2.20
2.26
3.11
f1 (ppm)
C4H9
O
N
O
OH
14a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169.08
165.46
163.78
136.93
134.28
131.72
128.89
126.04
124.54
120.94
120.46
102.08
39.79
30.12
20.38
13.83
f1 (ppm)
C4H9
O
N
O
OH
14a

Display Report

Analysis Info

		Acquisition Date	12/1/2021 11:52:42 AM
Analysis Name	D:\Data\new user data 2021\Dec-2021\1-Dec\Dr.M.Kapur-NO-01-11-II.d		
Method	tune_low_neg.m	Operator	RUCHI
Sample Name	NO-01-11-II	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
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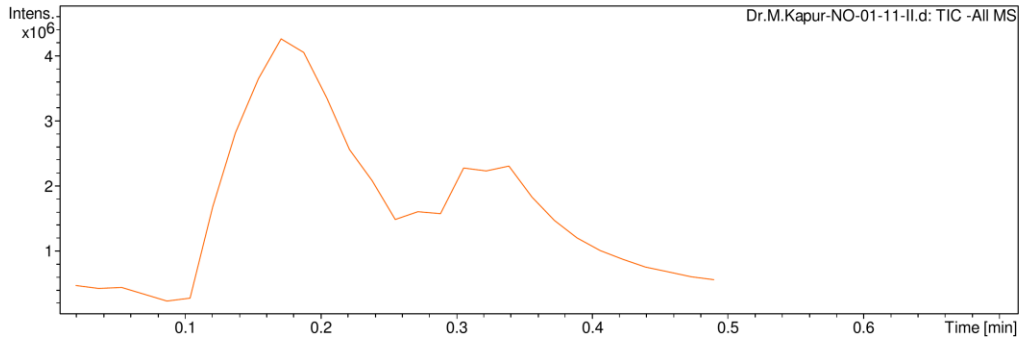
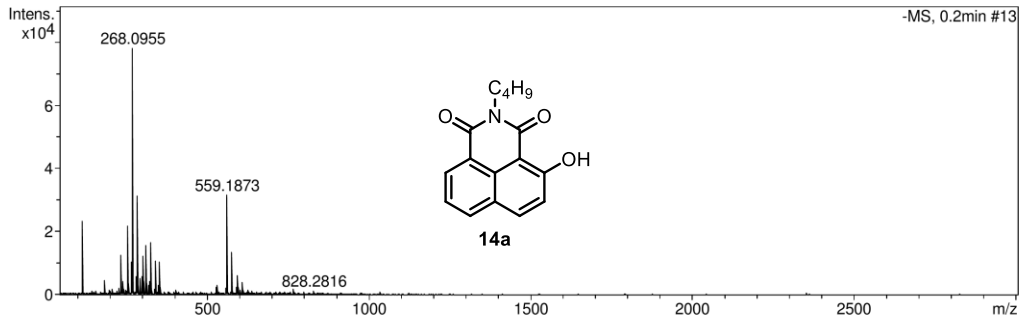
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste





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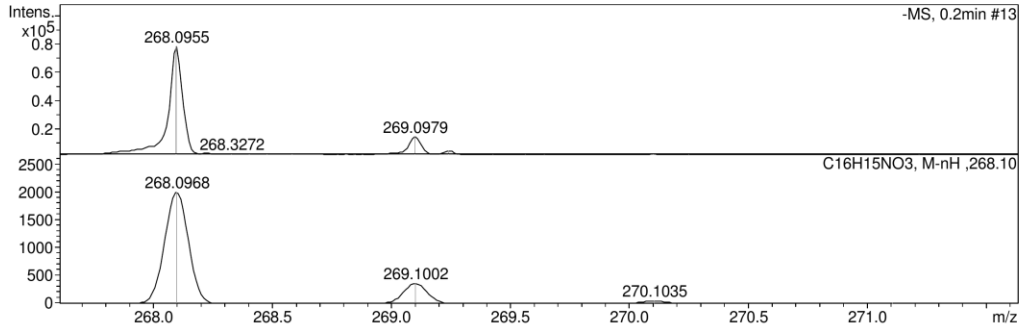

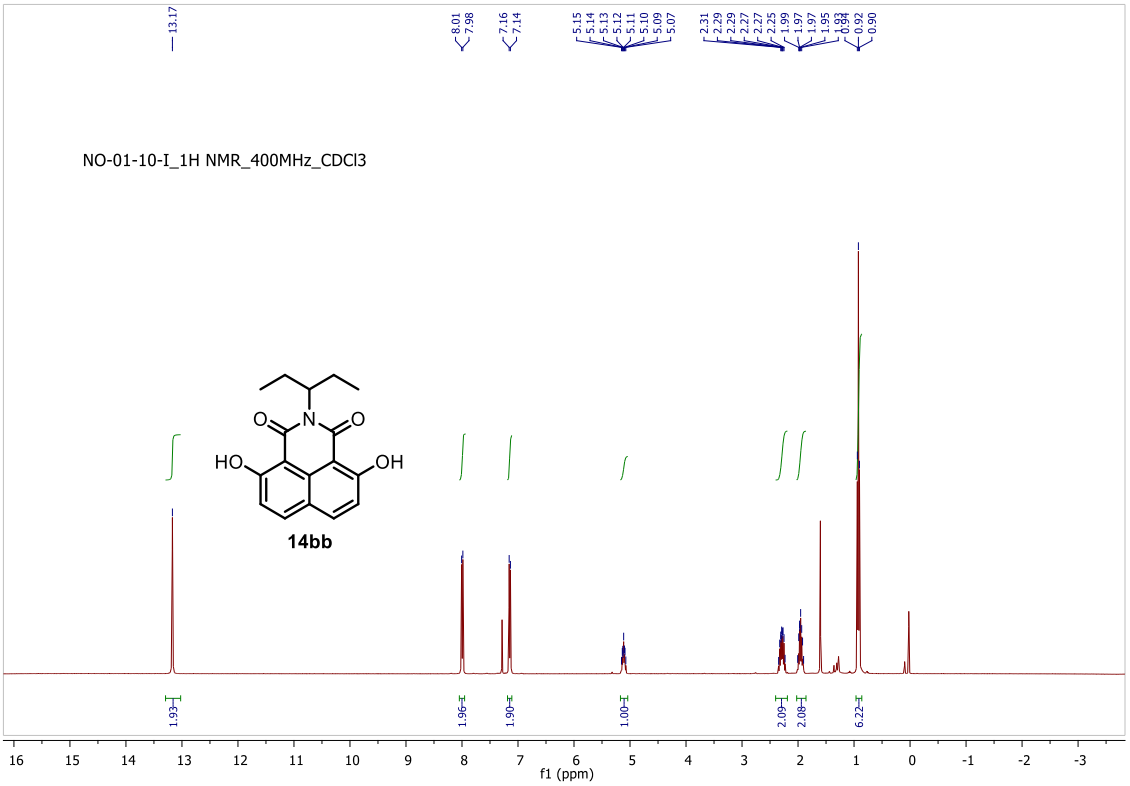
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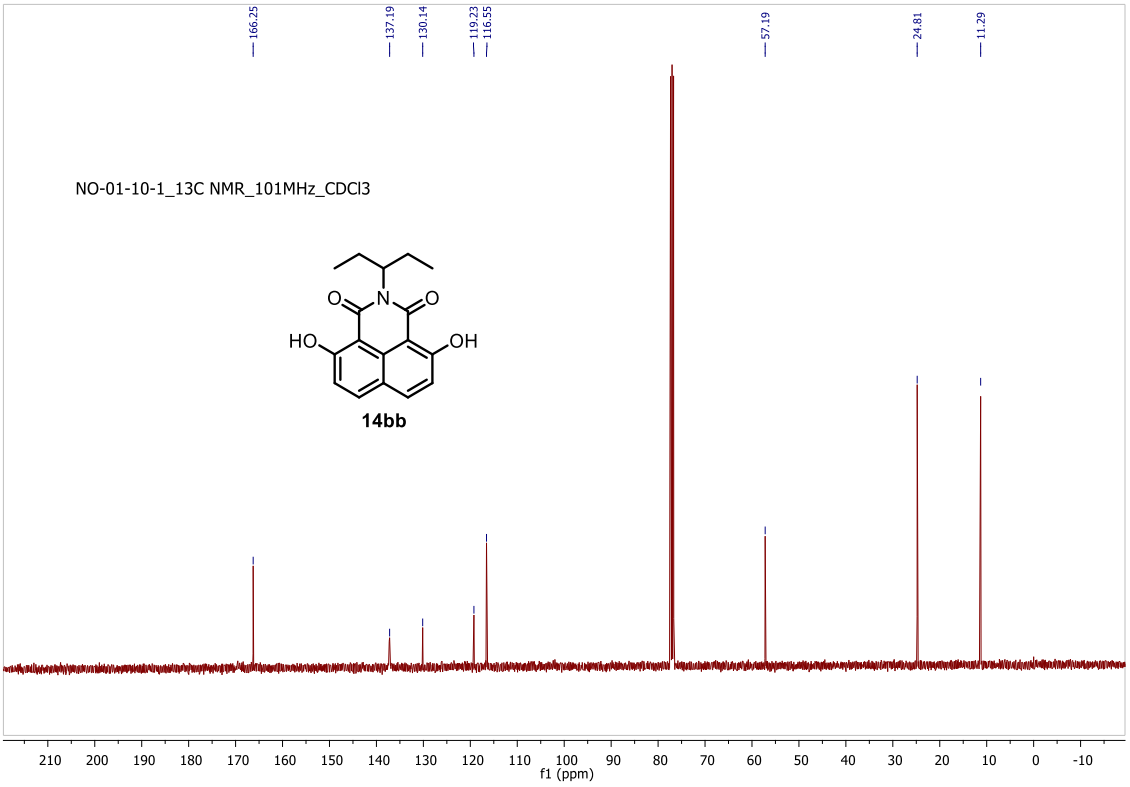
8.01
7.98
7.16
7.14
5.15
5.14
5.13
5.12
5.11
5.10
5.09
5.07
2.31
2.29
2.29
2.27
2.27
2.25
1.99
1.97
1.97
1.95
1.93
0.94
0.92
0.90
HO
OH
14bb
1.93
1.96
1.90
1.00
2.09
2.08
6.22
f1 (ppm)


NO-01-10-1_13C NMR_101MHz_CDCl3
166.25
137.19
130.14
119.23
116.55
57.19
24.81
11.29
HO
OH
14bb
f1 (ppm)

Display Report

Analysis Info

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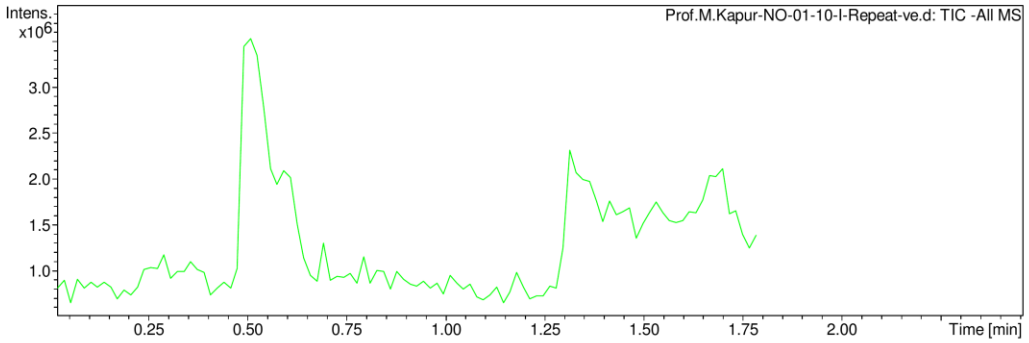
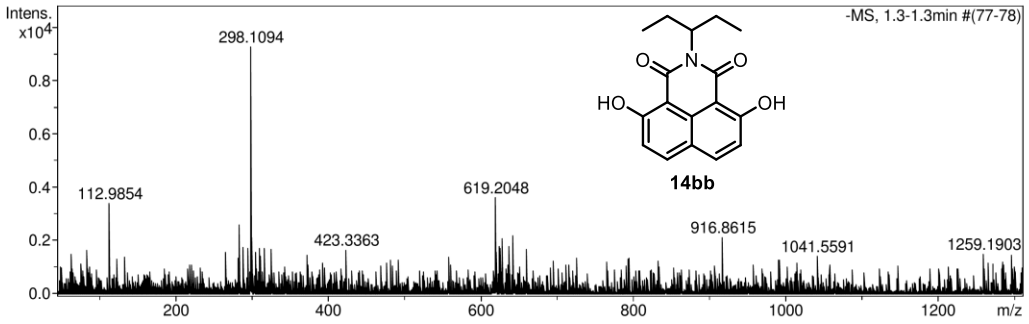
Analysis Name	D:\Data\new user data 2021\Nov-2021\29-nov\Prof.M.Kapur-NO-01-10-I-Repeat-ve.d		
Method	tune mix_low.New.021117.m	Operator	RUCHI
Sample Name	NO-01-10-I-Repeat-ve	Instrument	micrOTOF-Q II 10330
Comment			

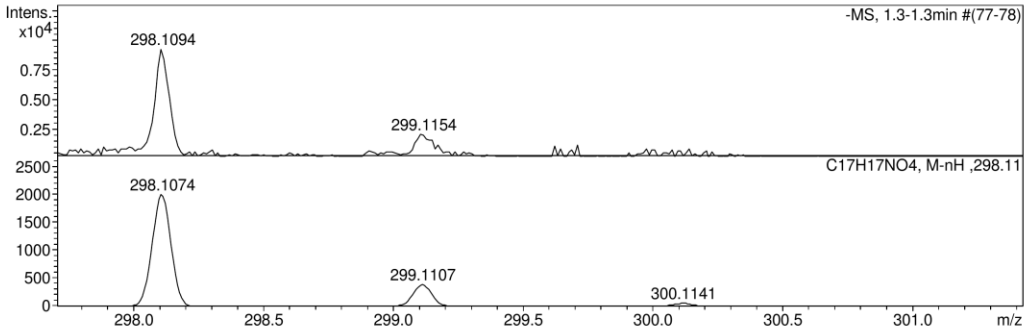
Acquisition Parameter

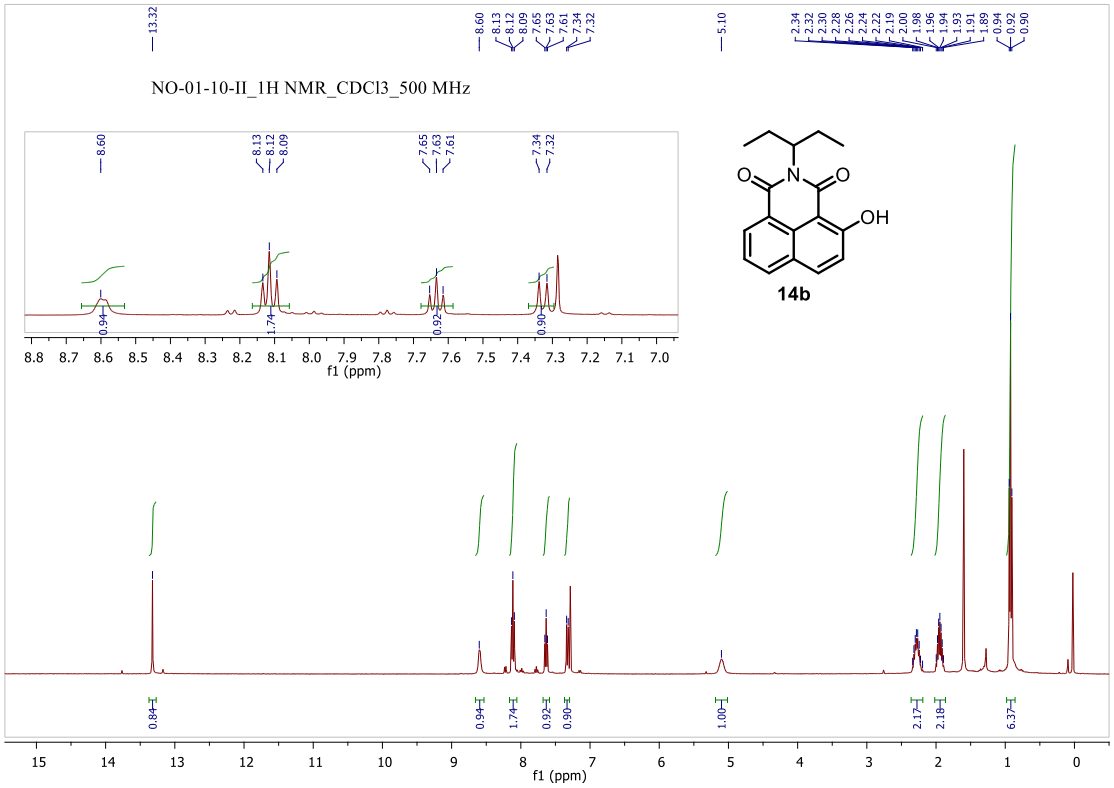
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Waste





14bb

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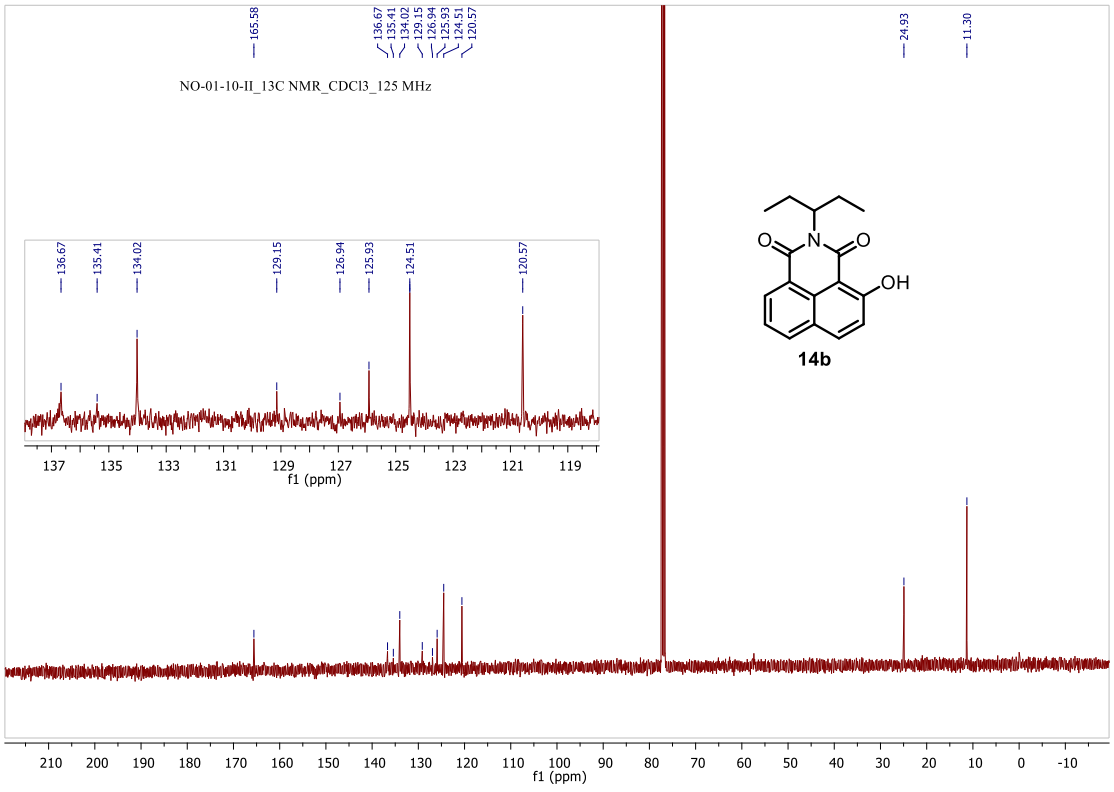
14b
f1 (ppm)


NO-01-10-II_13C NMR_CDCl3_125 MHz
14b
f1 (ppm)

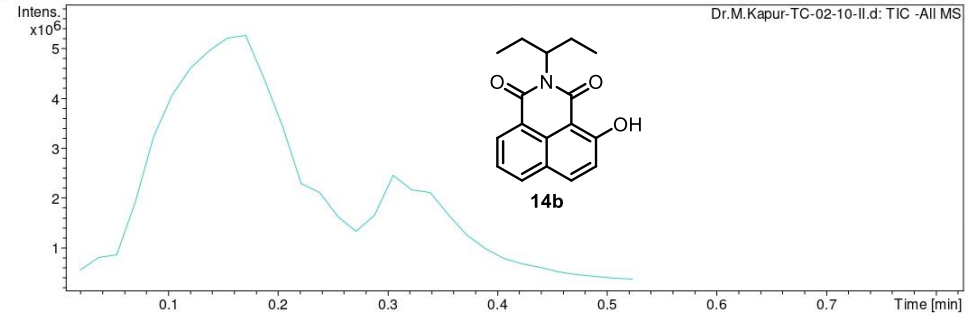
Display Report

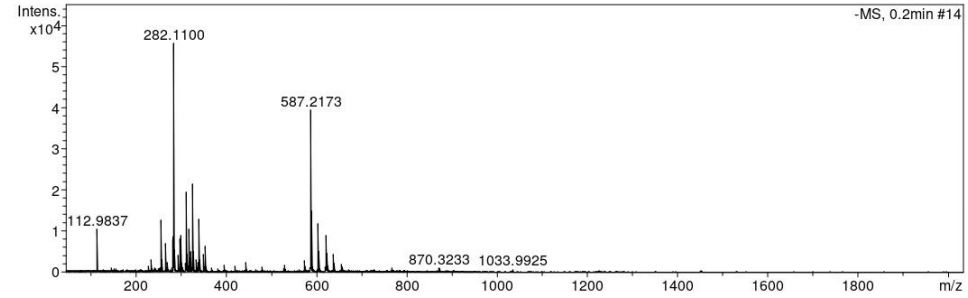
Analysis Info

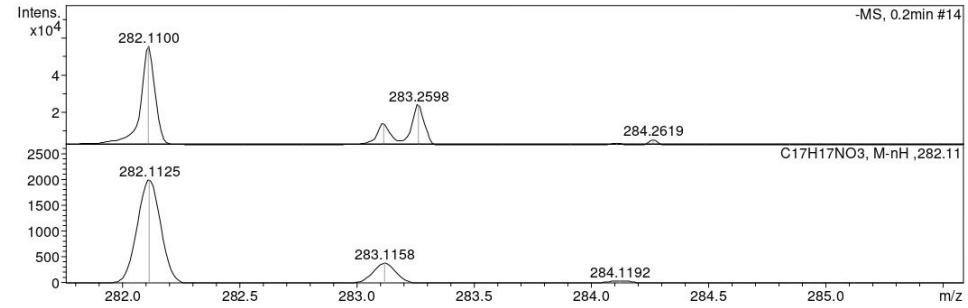
Analysis Name	D:\Data\new user data 2021\Dec-2021\1-Dec\Dr.M.Kapur-TC-02-10-II.d	Acquisition Date	12/1/2021 11:54:58 AM
Method	tune_low_neg.m	Operator	RUCHI
Sample Name	TC-02-10-II	Instrument	micrOTOF-Q II 10330
Comment			

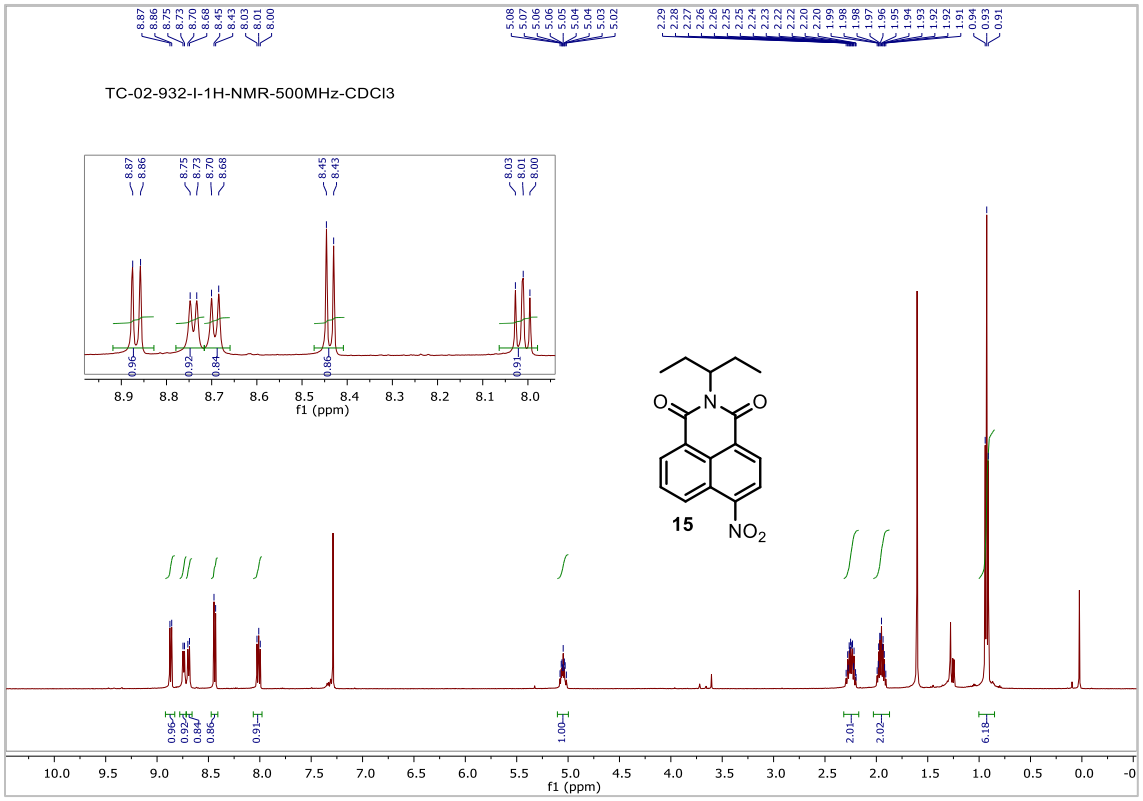
Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste





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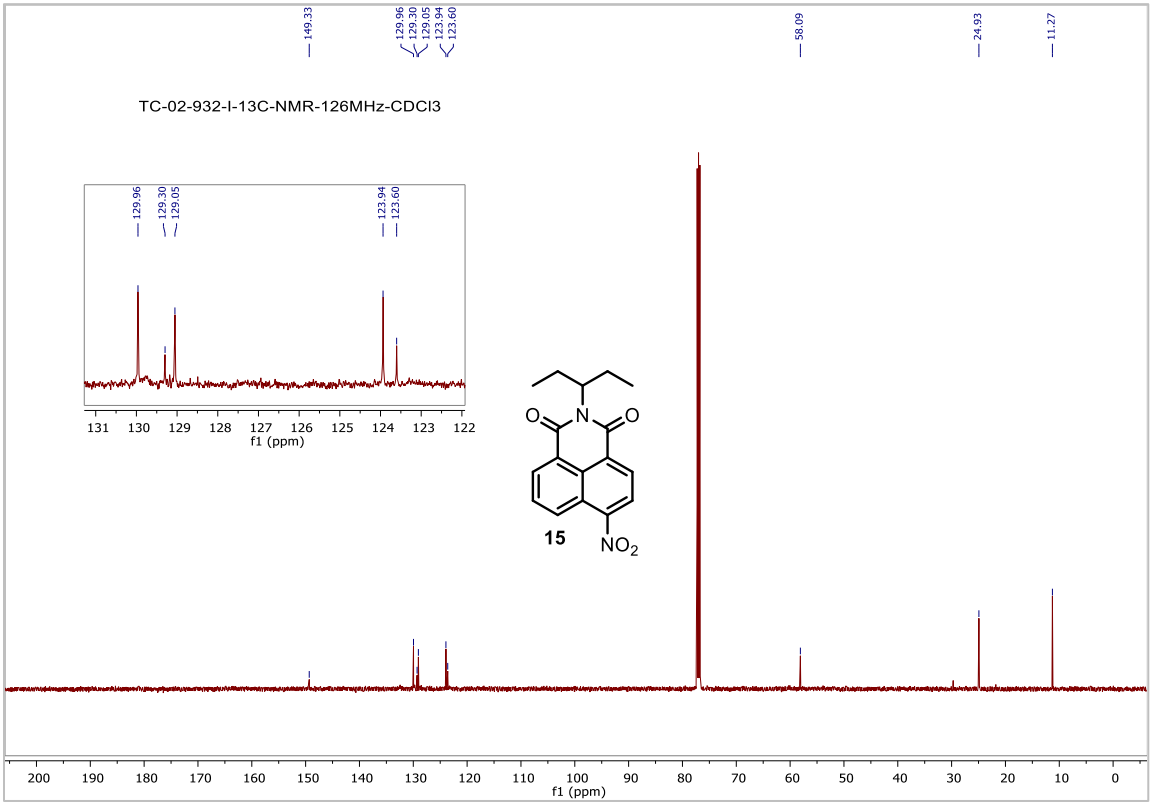
15
NO2
f1 (ppm)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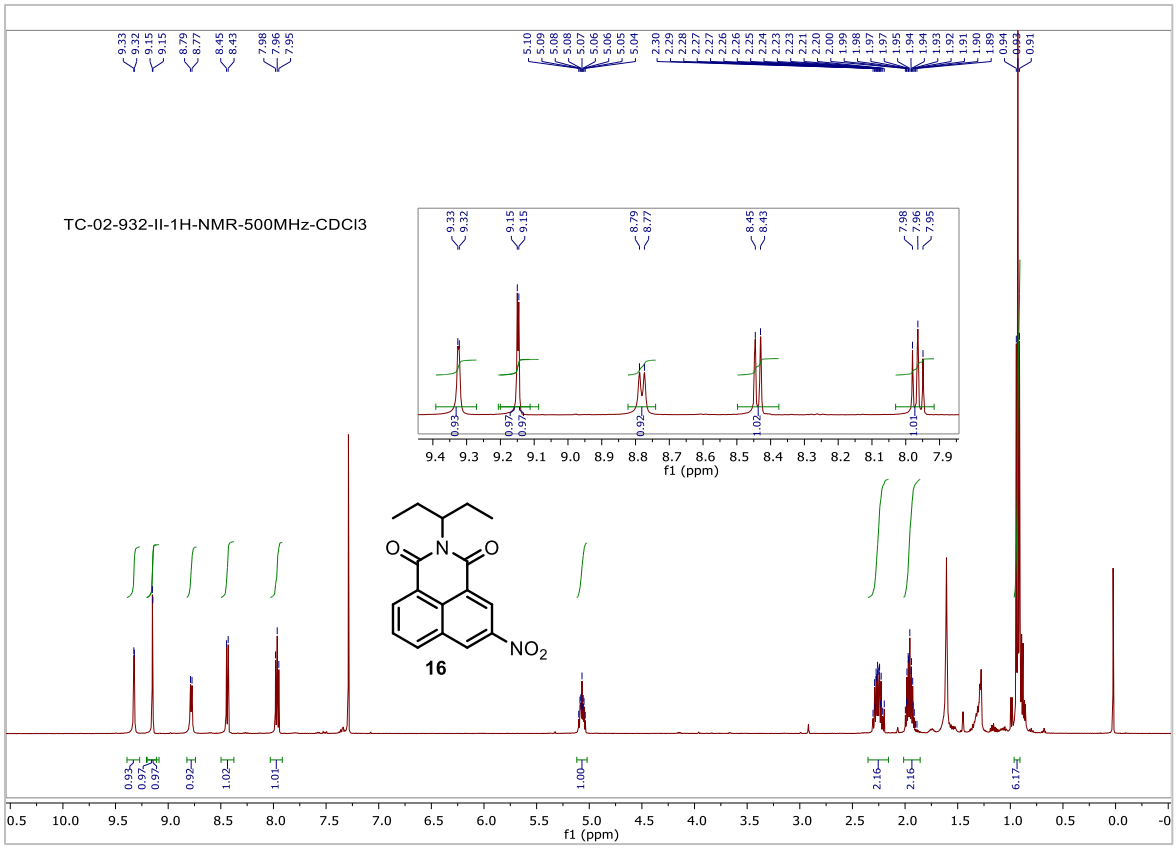
15
NO2
f1 (ppm)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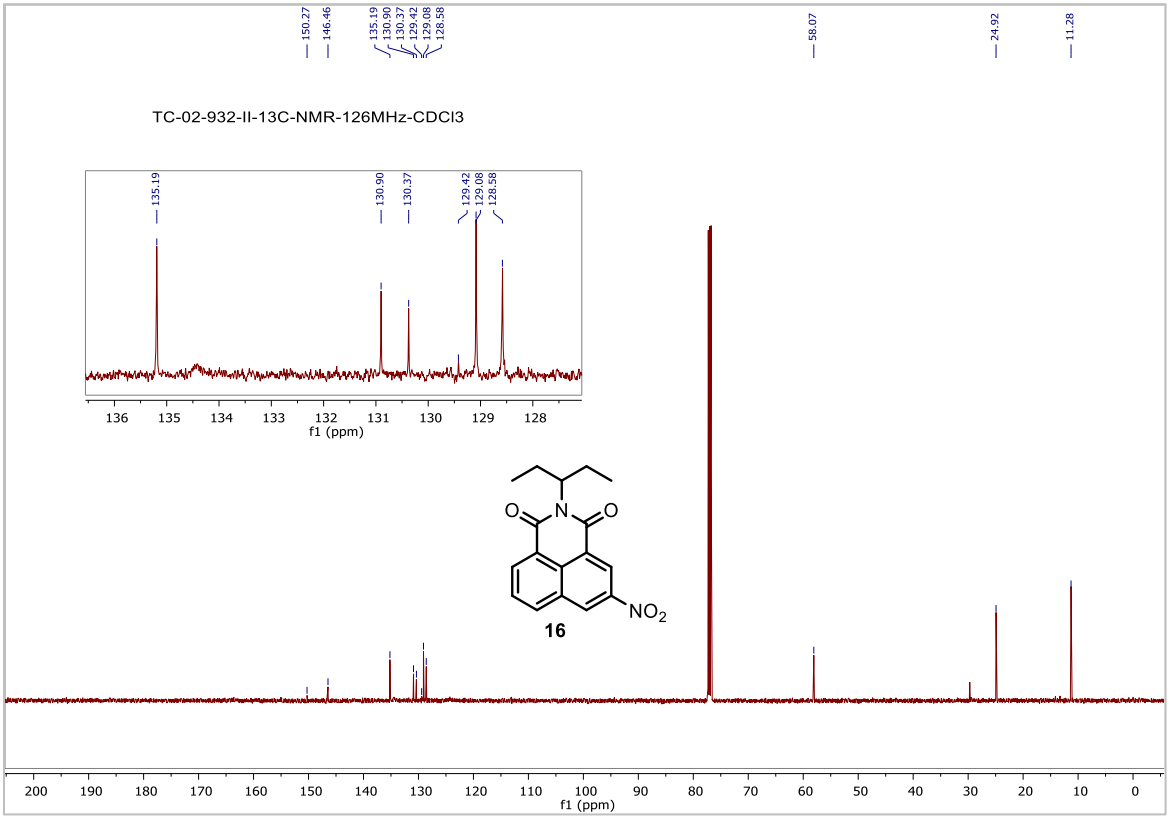
16
f1 (ppm)


TC-02-932-II-13C-NMR-126MHz-CDCl3
16
f1 (ppm)

Display Report

Analysis Info

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Method	tune mix_low.New.021117.m	Operator	Bruker
Sample Name	TC_02_932_II-R	Instrument	micrOTOF-Q 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
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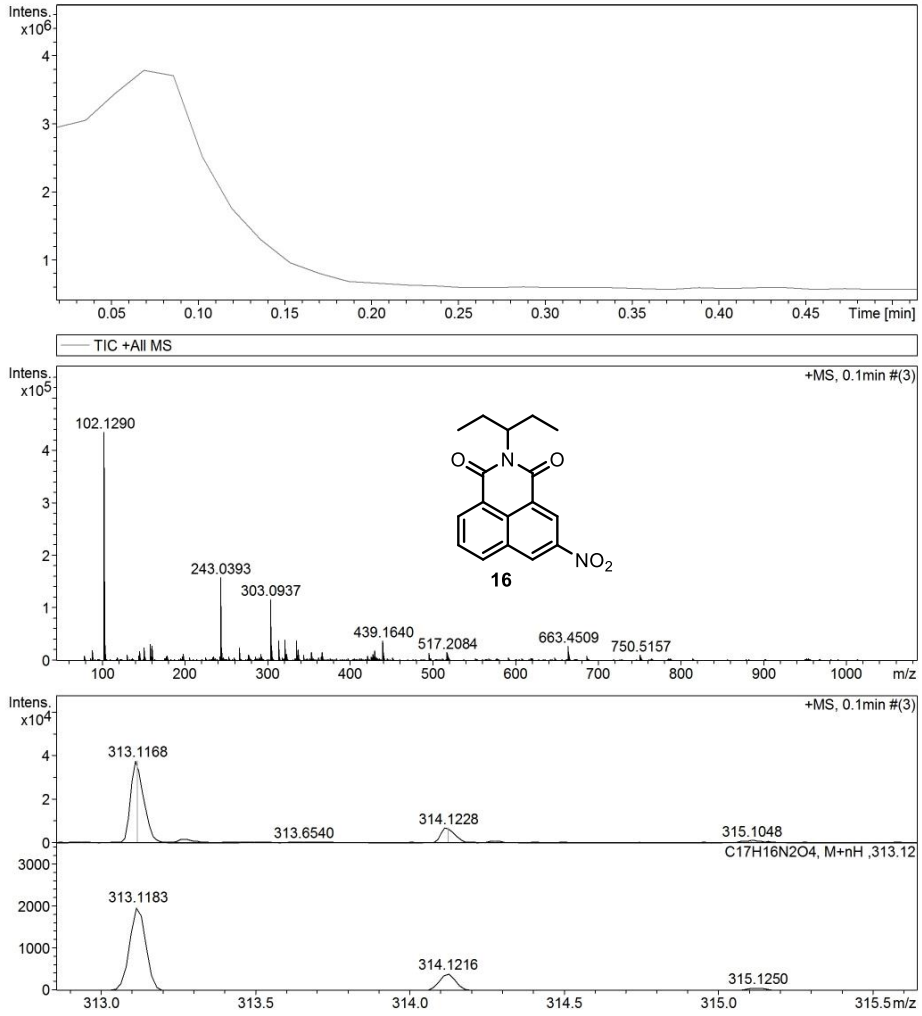
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Source



UV-vis Absorption and Emission Spectra:

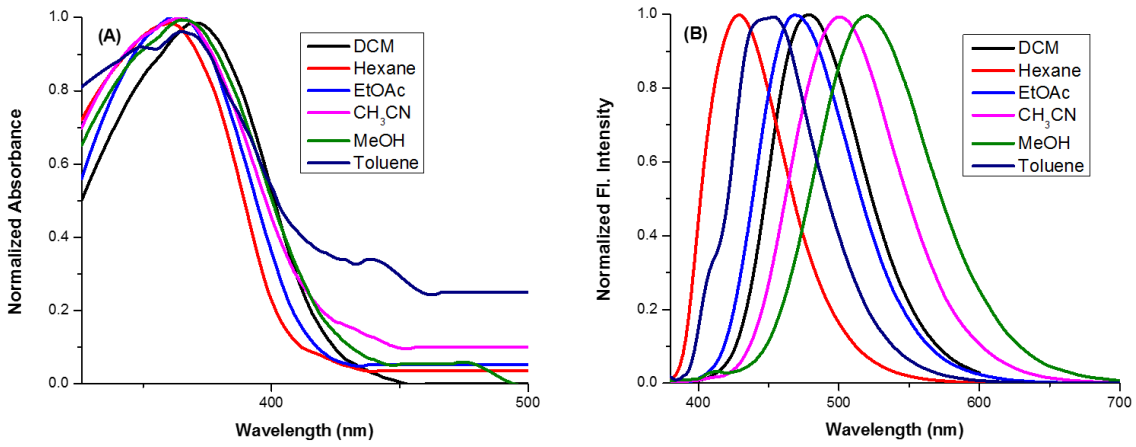


Figure S1. Normalized (A) UV-vis absorption and (B) fluorescence spectra of compound **8t** in DCM, *n*-Hexane, Ethyl acetate, Acetonitrile, Methanol and Toluene.

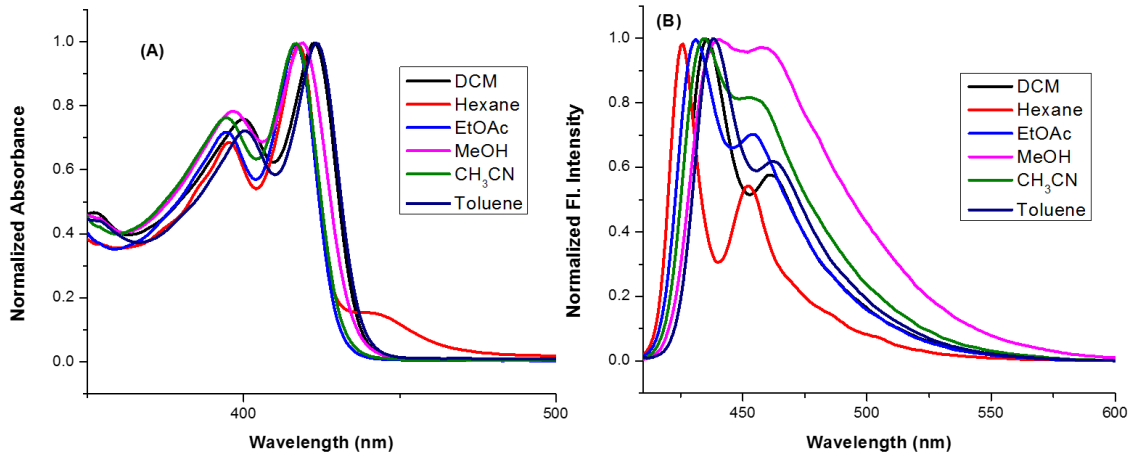


Figure S2. Normalized (A) UV-vis absorption and (B) fluorescence spectra of compound **10** in DCM, *n*-Hexane, Ethyl acetate, Acetonitrile, Methanol and Toluene.

Table S8: Optical properties of compound **8t** and **10** at the very dilute solution (2 μM) in six different solvents (dichloromethane, *n*-hexane, ethyl acetate, methanol, acetonitrile and toluene) having different polarity.

Solvents	**Compounds**			
	8t		**10**	
	λ_{abs}^{max} (nm)	λ_{em}^{max} (nm)	λ_{abs}^{max} (nm)	λ_{em}^{max} (nm)
DCM	370	478	423	436
Hexane	360	429	418	426
Ethyl acetate	363	468	416	432
Acetonitrile	364	500	419	435
Methanol	364	519	416	441
Toluene	364	454	423	439

X-ray crystallographic study of 2a:

Sample preparation: 5 mg of **2a** (yellow precipitate) was taken in a 10 mL beaker and dissolved in a minimal amount of methanol. Chloroform (5 mL) was added to the beaker along the wall. The beaker was capped loosely and kept at room temperature for slow evaporation. After seven days, a single crystal was obtained which was subjected to X-ray diffraction.

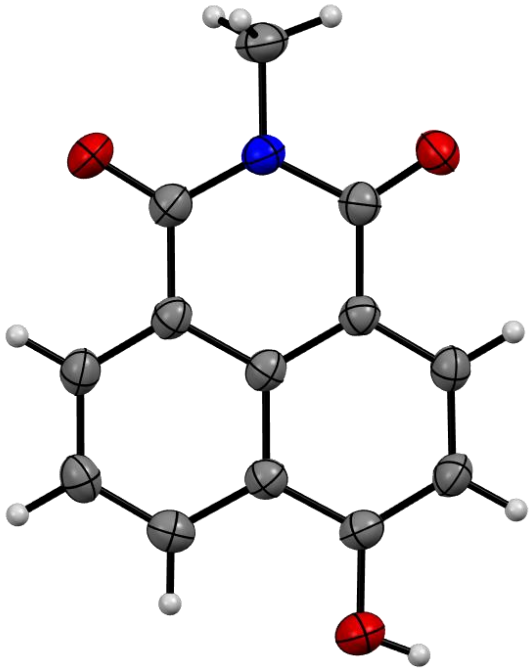


Figure 1: Crystal structure of **2a** (CCDC 2265977), showing thermal ellipsoid at 50% probability level.

2a (NO-01-71-P)

Table 1 Crystal data and structure refinement for 2a.

Identification code	NO-01-71-P
Empirical formula	$C_{2.6}H_{1.8}N_{0.2}O_{0.6}$
Formula weight	45.444
Temperature/K	185.00
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	3.8378(2)
b/Å	8.6367(5)
c/Å	29.4303(19)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	975.49(10)
Z	20
ρ_{calc}g/cm^3	1.547
μ/mm^{-1}	0.927
F(000)	473.8
Crystal size/mm^3	0.59 × 0.46 × 0.32
Radiation	Cu Kα (λ = 1.54178)
2Θ range for data collection/°	6 to 133.16
Index ranges	$-4 \leq h \leq 4$, $-10 \leq k \leq 7$, $-34 \leq l \leq 35$
Reflections collected	4526
Independent reflections	1661 [R_{int} = 0.0468, R_{sigma} = 0.0498]
Data/restraints/parameters	1661/0/157
Goodness-of-fit on F^2	1.046
Final R indexes [I>=2σ (I)]	R_1 = 0.0491, wR_2 = 0.1361
Final R indexes [all data]	R_1 = 0.0529, wR_2 = 0.1390
Largest diff. peak/hole / e Å^{-3}	0.38/-0.25
Flack parameter	0.3(2)